Corrosion

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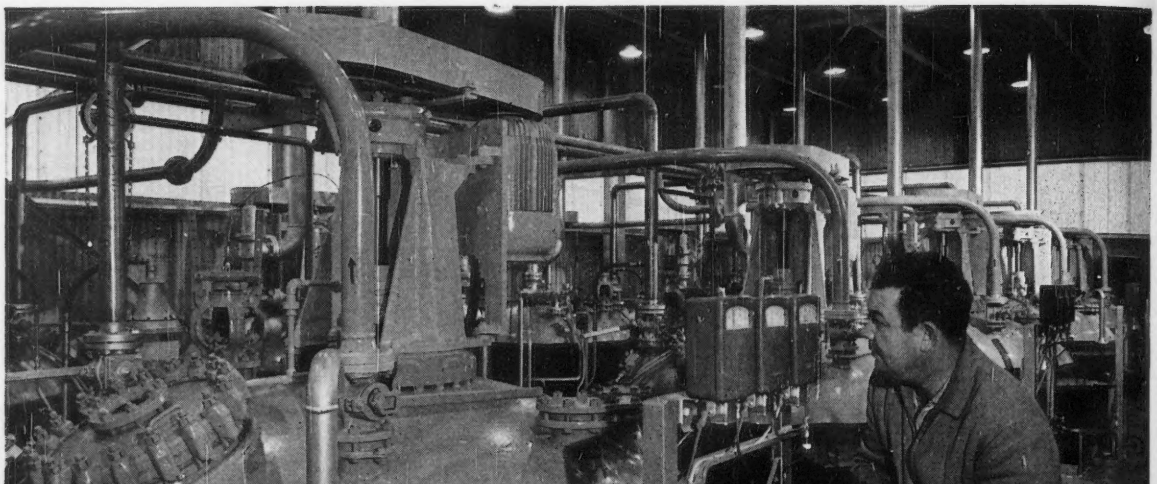

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This Month in Corrosion Control . . .

TRIFLUOROCHLORETHYLENE's physical properties are discussed with regard to many types of application for this versatile plastic in an article beginning on Page 14. Also covered are some of the characteristics of fluorocarbon copolymer coatings, and their resistance to about 150 corrosive chemicals and solvents.

HOW COATINGS react to the hot, humid marine environment of a chemical plant on the Texas Gulf Coast is the topic of an article beginning on Page 20. This is the third and fourth parts of a comprehensive discussion of the coatings problem at a chemical plant, and how it was solved.

YOU CAN INVESTIGATE the comparison's between some structural plastics and metals in corrosive environments in an article starting on Page 25. Some of the cost considerations important in specifying non-metallic pipe are given also.

TWENTY-THREE COMMITTEES are now affiliated with NACE's T-7 Corrosion Corrinating Committee following the addition of groups at Chicago and Detroit.

WILLIAM C. KINSOLVING, president of Sun Pipe Line Company will address a luncheon meeting of corporate representatives of NACE at Dallas March 17, during the annual NACE Conference there.

NO WHEELS—LESS METAL. Is This the Car of the Future? Some aspects of the new look in personal transportation are shown on Page 39.

RUSSIAN PERIODICALS carrying large volumes of information on corrosion control are named on Page 46. Also given are names and locations of publications listing English translations from these and other leading Russian magazines.

IT IS NOT ALWAYS EASY to measure underground currents and it is even more difficult sometimes to discover their source. A long-time worker on underground structure corrosion problems defines some of the ways in which the source of these currents can be ascertained in an article beginning on Page 87.

MILD AND STAINLESS STEELS used to handle oxidizing acids at temperatures up to 500 F can be protected with negligible electrical currents using a system developed by a research team at the Continental Oil Company's Ponca City, Oklahoma laboratories. Theoretical aspects, reports on application in an operating plant and some estimates of possible uses of the method in the chemical industry are covered in detail in three articles beginning on Page 91.

LABORATORY SCREENING of inhibitors intended for use in sour oil and gas wells is an essential step in their selection for actual service. NACE's T1-K-1 Committee on Sour Crude Inhibitor Evaluation proposes a standard static laboratory test in a report beginning on Page 107.

MORE THAN 100 COMPANIES using high silicon cast iron as inert anodes in cathodic protection systems have reported their experience to NACE T-2B-4 on High Silicon Cast Iron Anodes. This experience is tabulated beginning on Page 109.

DUCTILE VANADIUM is less noble than stainless steel and copper and more noble than aluminum, magnesium and steel in synthetic sea water. Other interesting fundamental information about this interesting metal also will be found in an article beginning on Page 114.

TWENTY-SIX STRUCTURAL and cladding alloys were tested in 1000-2000 psi carbon dioxide at 1100-1740 F to give data presented in an article beginning on Page 117.

SUITABILITY OF INCONEL for use in nuclear power plant applications is described in an article beginning on Page 123.

CORROSION RATES often are functions of velocity, among other things. H. R. Copson of Inco's Bayonne research laboratory covers many of the essential facts about this phenomenon in his article in this issue. Turn to Page 130.

ZIRCONIUM ALLOYS are being used with greater frequency in many highly corrosive environments. Results of tests made on two patented alloys are given in an article beginning on Page 137.

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AN EDITORIAL

Research Chickens Come Home to Roost

SOMETIMES an opportunity comes to bring home strongly the truism that "research chickens come home to roost." Such an opportunity arises because of the recent recognition by an oil company research team that a 50 year old discovery by a man doing so-called "pure" research had important commercial possibilities. The discovery was made by Flade in 1911 that iron in sulfuric acid reached a passive state when, after having been made the anode in an electrical circuit, high values of current were passed through it. Flade's diagram also showed this passivity continued under certain clearly defined electrical conditions.

The key that opened this discovery to practical use was an electrical device that kept the iron surface under the electrical conditions which made it passive. This device, after numerous tests, has been put to work by this research team protecting steel from attack by oxygen-bearing aqueous solutions with a very nominal outlay for equipment and at a modest energy cost.[1, 2, 3]

This may have far-reaching effects in the selection of materials for and the design of vessels and piping systems handling these solutions. For example, carbon steel may be substituted for stainless steel vessels and piping in many process streams including sulfuric acid. It is possible also that further investigation may reveal as yet unsuspected variations of the initial discovery which will extend its application.

An analogous although not quite so dramatic use of discoveries in the fundamental sphere is the growing awareness and use of the "Pourbaix Diagrams" as a device for preliminary estimates on the reaction of a material to its environment. Awareness of Mr. Pourbaix's work,[4] first published in CORROSION in April, 1949 (itself a translation of a 1946 article published in Paris) is growing. Many references are seen to his method of plotting the corrosive characteristics of a system.

The discovery that a palladium addition to titanium improved its resistance to some boiling solutions of reducing acids (CORROSION, June, 1959, Page 81) was foreshadowed to some extent by the experiments and empirical results reported by many earlier workers on surface states. Some of these fundamental discoveries were reported by Tomashov,[5] and others. It took an alert and inquiring mind to see the practical application of these hypotheses and skillful investigation to prove the reliability of the conclusions.

Instances of this kind could be reported almost endlessly. It does not follow thereby, however, that all practical discoveries come from pure research although it is true that many apparently unoriented discoveries have been recognized later as having practical applications. Many more will be so recognized.

This underlines the importance of having accessible records available on the side discoveries of directed as well as those made in undirected research. Science and mankind gain from the free revelation of these facts.

1. J. D. Sudbury, O. L. Riggs, Jr., and D. A. Shock. Anodic Passivation Studies. *Corrosion*, 16, 47t (1960) Feb.
2. D. A. Shock, O. L. Riggs, Jr., and J. D. Sudbury. Application of Anodic Protection in the Chemical Industry. *Corrosion*, 16, 55t (1960) Feb.
3. O. L. Riggs, Jr., Merle Hutchinson and N. L. Conger. Anodic Control of Corrosion in a Sulfonation Plant. *Corrosion*, 16, 58t (1960) Feb.
4. M. Pourbaix. Corrosion, Passivity and Passivation From the Thermodynamic Point of View. *Corrosion*, 5, No. 4, 121-133 (1949) April.
5. N. D. Tomashov. Methods for Increasing the Corrosion Resistance of Metal Alloys. *Corrosion*, 14, No. 5, 229t-244t (1958) May.

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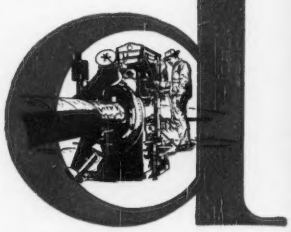

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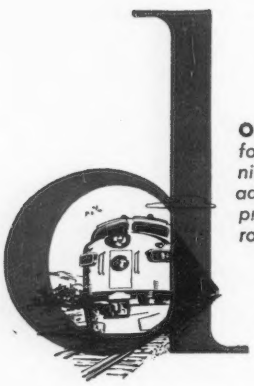

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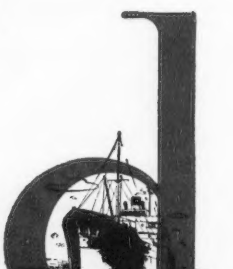

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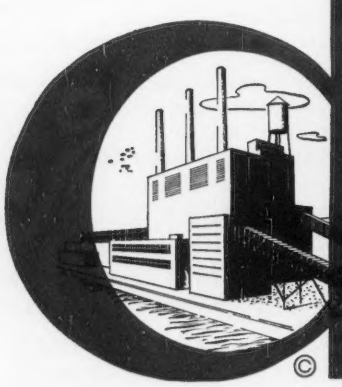

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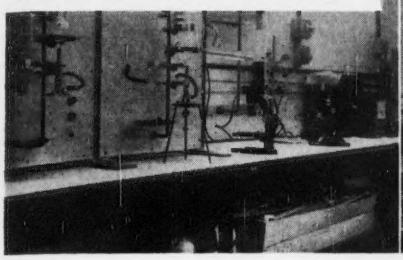
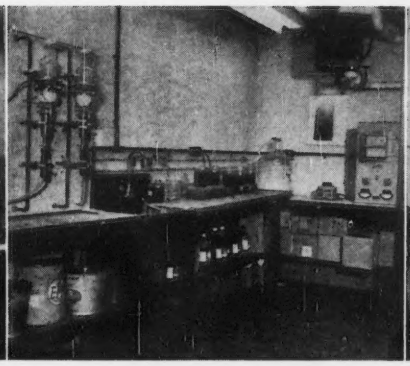


FLUORINE APPARATUS used for fluorine determinations to set solution strengths is shown on the left. Titration table and miscroscopy equipment are at the right.



TWO UNITS on the left are used for determining corrosion rates for hot wall effects as encountered in reboilers or evaporating surfaces. On the right is a Burrell electrical furnace for heat-treating metal specimens to determine heat effects on corrosion.

HYDROFLUORIC ACID SCREENING corrosion test (in polyethylene containers) is shown above. Uranium salts tests (in the larger pail to the right) was conducted in which a sealed stainless beaker lined with polychlorotrifluoroethylene was held at temperature in an oil bath.

Functions of the

Materials Engineer in the Chemical Industry*

Abstract

Rapid advances in materials and corrosion and the resulting wealth of knowledge obtained are making possible a more scientifically constructive approach to industry's problems. Many companies now have metallurgical, corrosion or materials engineers employed full time and are obtaining benefits not only in longer equipment life but also in improved process efficiencies. This article shows how the General Chemical Division of Allied Chemical Corporation has set up within a centralized engineering department a Materials engineering section. Functions of this section are explained not only within that department but with all other related company departments. 8.8.1

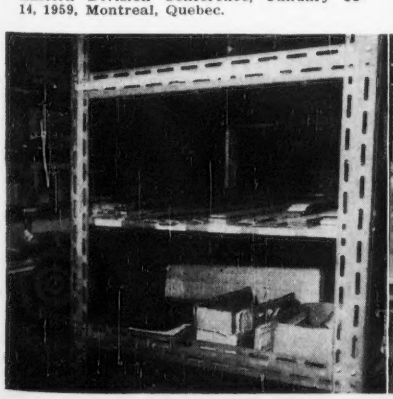
RAPID ADVANCES in the development of new materials during the past two decades have created the need for a specialist in the materials engineering field. Optimum use of new materials is possible only when a materials specialist employed by the user can apply materials by working with suppliers and his own company's engineers. This is true because the corrosive conditions and process variables found in industry are so varied that suppliers of materials cannot possibly know of all the potential uses for their products. The chemical industry, for example, is a consumer of these materials and has applications for everything from paints and plastics to the high alloy and rare metallic elements.

The General Chemical Division of Allied Chemical Corporation has titled its materials specialist as "Materials Engineer." Although this is not a new title, the broad knowledge and background necessary for this engineer to be able to specify engineering materials for chemical processes is a new concept. The materials engineer should be either a mechanical or chemical engineer with some graduate or undergraduate work in metallurgy, welding, fabrication, plastics, ceramics, corrosion and equipment design. The diversity of materials and their uses necessitates specialization in fields such as paints, plastics, ceramics or metals.

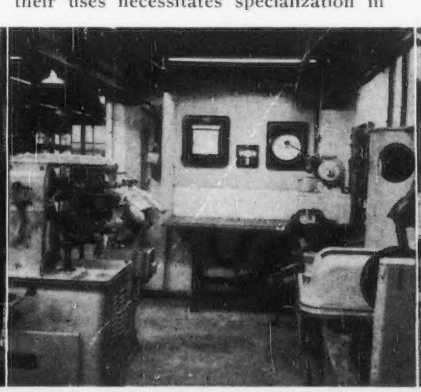
Although the General Chemical Division was using special materials before World War II, the need for materials specialization was not fully recognized until 1945. War-time shortages and resulting substitutions proved that a better understanding of the newer materials was necessary if repeated failures were to be avoided. General Chemical embarked on a material and corrosion control program in November, 1945. A result of this is the present Materials Engineering Section located in the division's Central Engineering Department. This section includes eight full-time men engaged in materials and corrosion

(Continued on Page 10)

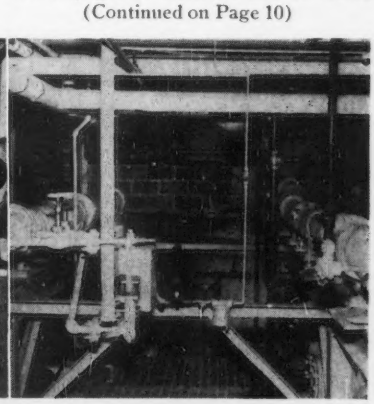
★Revision of a paper titled "The Materials Engineer in the Chemical Industry" by W. H. Burton, Allied Chemical Corp., Camden, N. J., presented at the Canadian Region Eastern Division Conference, January 12-14, 1959, Montreal, Quebec.



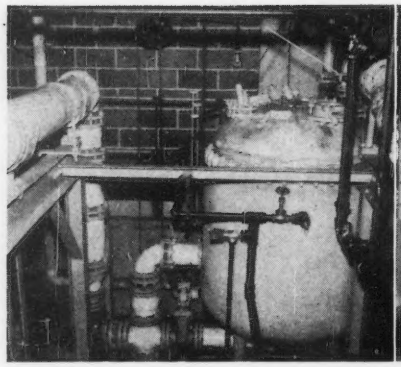
RACKS for identified alloy specimen stock are shown. A variety of materials is required to conduct corrosion tests. Non-metallic specimens are stored on racks shown in the rear.



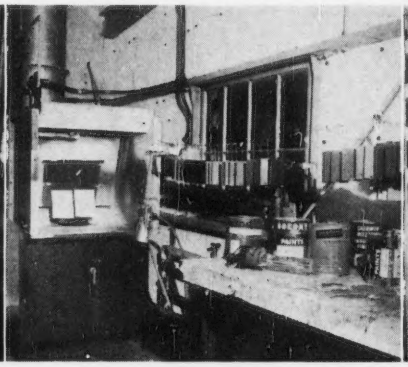
CORROSION TEST SPECIMENS are prepared in this machine shop section.



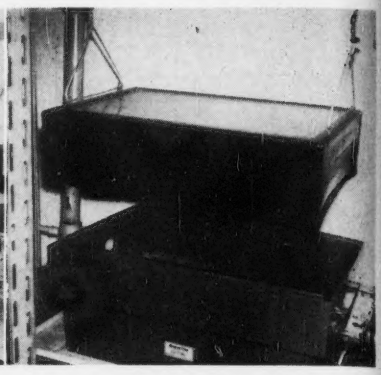
VELOCITY TESTING EQUIPMENT, shown above, includes a hairpin loop through which a corrosive solution is pumped at constant velocity and temperature. The loop consists of six-, five,- and four-inch pipe sections connected in series with specimens mounted in each. One pumping thus develops three velocities.



IN VELOCITY TESTS, the corrosive solution is pumped from and returned to this jacketed, glass-lined kettle.



TEST SPECIMENS are spray painted and dried in this section of the laboratory.



PAINT SPECIMEN PANELS are exposed on trays in this spray box under simulated atmospheric exposures.

Materials Engineer—

(Continued From Page 9)

work. A well equipped laboratory is part of the section.

Functions Within the Department

Figure 1 shows how this section functions in the engineering department. To successfully complete the design of a new plant, a project engineer must call on the various engineering service groups and then coordinate the information for the project. This is indicated by the dash lines in the figure. The Materials Section functions as do the other service groups. When a project is scheduled, the project engineer discusses the job with the various groups, and cooperating engineers are assigned as required. In this manner, a materials engineer is assigned and can review the job for materials and corrosion requirements in advance so that tests can be begun if existing data or experience is insufficient. Recommendations are made to the project engineer; drawings, bills of material or specifications are reviewed as required to insure the material recommendation and to design corrosion out of the process.

The full lines in Figure 1 show how the Materials Section works with all the other service groups to supply their needs for materials information. Close cooperation with the Process Section is maintained to keep abreast of process variables for evaluating their effects on materials. The Power Section frequently needs information pertaining to wells, underground pipe protection, water systems, boiler corrosion, etc. Both the Electrical and Instrument Sections require materials knowledge for conduit, insulation, corrosion proof windings, instrument internals and piping.

The necessity for more corrosion proof structures is understood. The Civil Engineering Section is directly concerned with this and with such items as concrete mixes, concrete protection and waterproofing of building walls.

There is a constant need to review specific drawings for materials design, comments and drawing notes in the drafting room so that bills of materials will be more complete.

In the Equipment Design Section, materials are considered with respect to their end use, and it is most important that designs be studied for proper fabrication, welding techniques and possible heat treatments to prevent adverse effects resulting from manipulations during fabrication stages.

Fire Protection and Safety call on the Materials Section frequently for recommendations on fire resistant materials, corrosion resistant cables and slings, spark-free materials, resistant color paints for safety zoning and others.

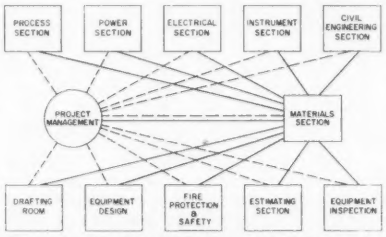


Figure 1—Function of the materials section in the engineering department.

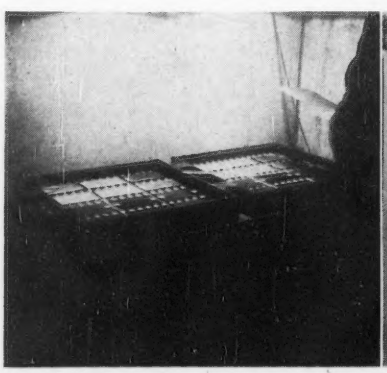
Although estimating is not a design function, there is constant need to check current costs of materials and fabrication. Cooperation with the Equipment Inspection Section is imperative to insure that materials used in fabrication are properly identified and to indicate to inspectors precautions required in operations such as forming, rolling, welding and heat treating. A review of these functions shows that no operations are possible without the use of materials and, with the rapid development and introduction of new types of materials, know-how is becoming increasingly important.

Functions With Other Departments

Functions of the Materials Section in General Chemical do not stop with the Engineering Department. Nearly all other company operations require its services as shown in Figure 2. Each of these departments is in direct contact with the Materials Section. Requests for information are filled from available data and experience; if necessary, laboratory or field tests are conducted.

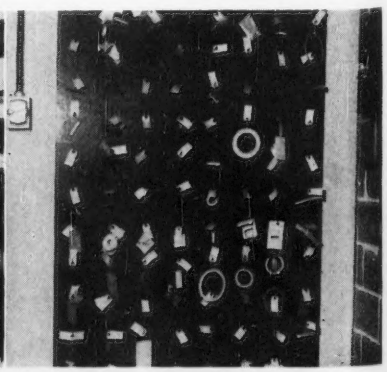
When equipment breakdowns occur, the assistance of the Materials Section is often requested by Production and Maintenance. At such times, the materials engineer must make field diagnosis of the type of failure to determine if it be stress corrosion cracking, knife line

(Continued on Page 12)

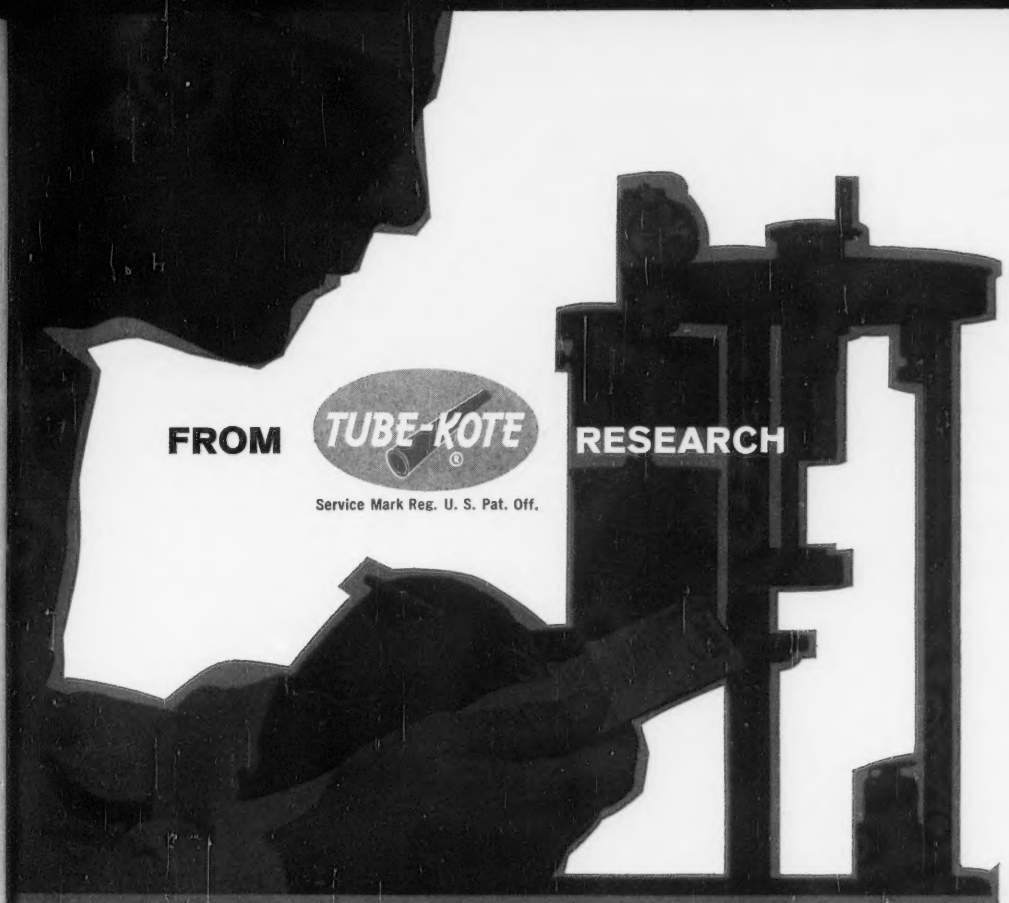


SPECIMEN PANEL RACKS are exposed to ultraviolet light for given periods to complete simulated exposure cycles. Panels are returned to the spray box and go through repeated cycles until failure.

EQUIPMENT FAILURES in the field are properly identified on tags and stored in the "Morgue" for reference.



PUMP PACKINGS and gasket materials standards were evaluated and placed on this board for ready reference because of difficulties experienced.



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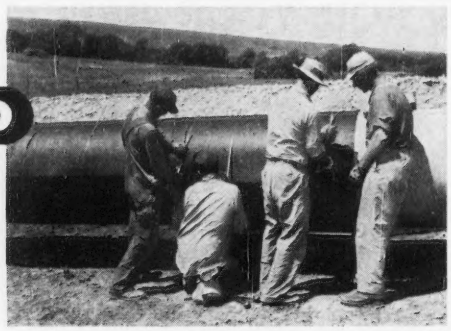

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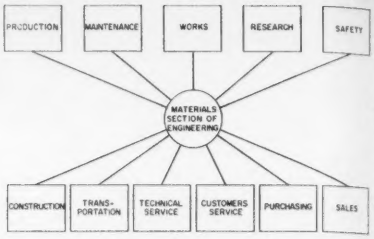


Figure 2—Function of the materials section in company operations.

Materials Engineer—

(Continued From Page 10)

attack, etc., or just general corrosion. He must then determine whether the cause was operational, improper material, faulty design or fabrication. Because most shutdowns are of an emergency nature, the materials engineer frequently makes re-design recommendations on the spot and defines specifications for repairs to meet code and safety requirements as necessary.

Some repairs require process changes such as variations in flow rates and solution strengths or reversal or direction of flow. While these changes are being handled from the field, the materials engineer remains in close contact with Central Engineering, Production and Maintenance so that all departments will be kept abreast of developments.

Transportation, in like manner, may have difficulties with tank cars, barges or truck trailers and require assistance as to fatigue, corrosion or design failures so that repairs can be made to satisfy governmental or other code requirements. Other departments shown in Figure 2 use the Materials Section to satisfy their particular needs.

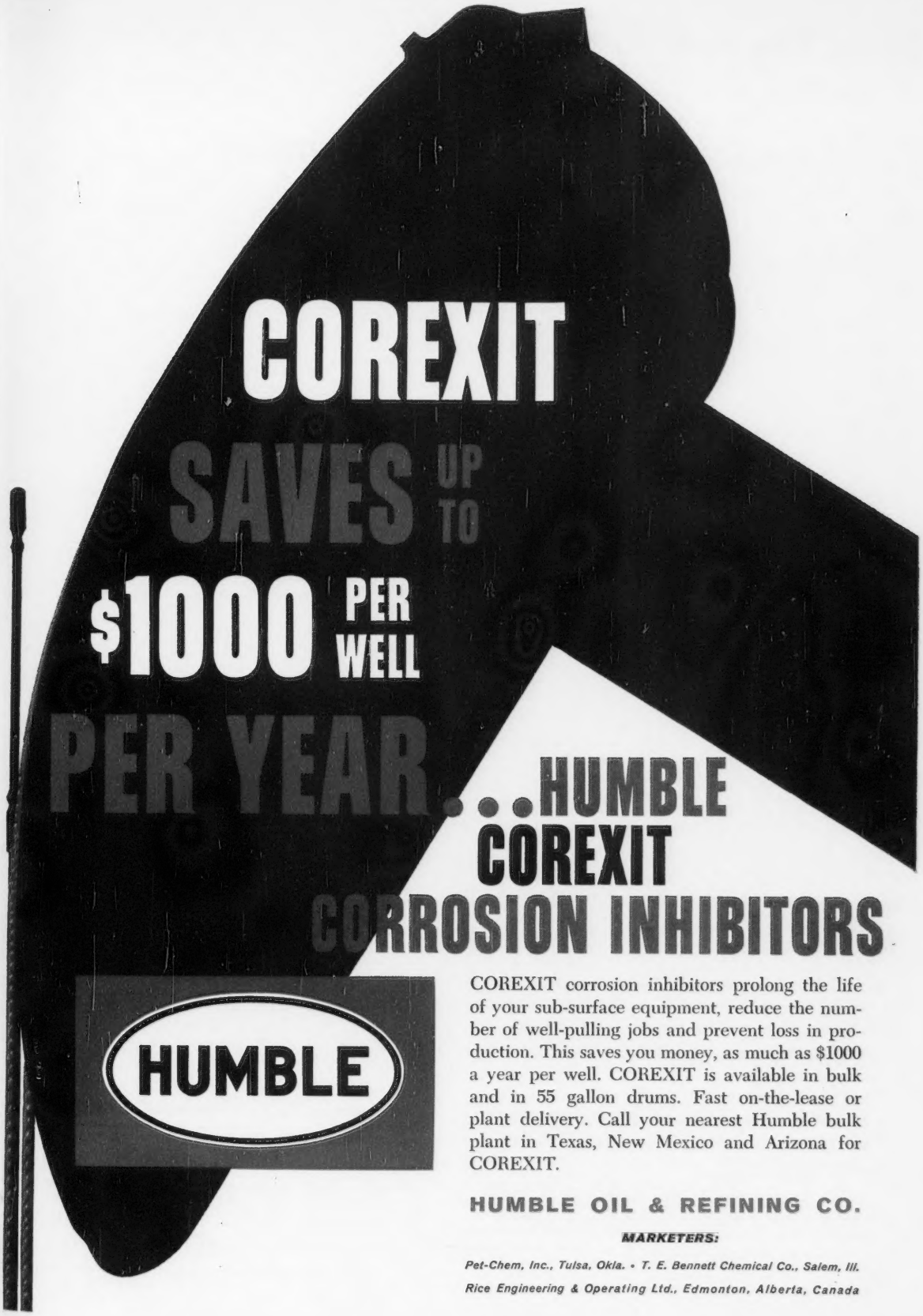
Corrosion Committee Organized

The original program started in 1945 included establishment of a Corrosion Committee so that close cooperation with other departments would be possible. This committee includes a representative from each of the most interested departments. Meetings are held regularly or on special call to set priorities on investigations or tests for major problems. The Materials Section then correlate these requests with other work required by engineering projects. A corrosion request or a materials investigation is initiated by a member of the committee and, with the committee's approval, is forwarded to the Materials Section. Installations resulting from such a test program are followed with field apparatus service records for their useful life or a maximum of ten years to correlate tests with actual service.

Laboratory facilities which have been provided to make materials tests and investigations are shown in the illustrations.

Conclusions

Advances made in corrosion engineering, coupled with new materials developments, have given the materials engineer additional tools to work with. Adequate facilities to use these new tools and to evaluate new materials should be provided. Along with facilities, he must have time and money for original study and for routine testing. Given company-wide cooperation, he can make a significant and outstanding contribution of saved man-hours and dollars for his company.



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Some Physical Properties of the Trifluorochlorethylene Polymers*

Abstract

Discusses physical properties, chemical resistance, compressive strength, dielectric properties and water adsorption of trifluorochlorethylene polymers. Tabular data give chemical resistance and dimensional stability of these polymers. Also gives properties of high molecular weight fluorocarbon resins and lists reagents resisted by fluorocarbon greases. 6.6.8

TRIFLUOROCHLORETHYLENE POLYMERS are available as a thermoplastic molding powder, as dispersions of this molding powder, as lacquers, resins, elastomers and as oils, waxes and greases.

Properties

These polymers are produced as high and low density molding powders ranging in molecular weight from 75,000 to 110,000. They are white and amorphous with a specific gravity of 2.1. In the molded form, they can be used over a range from the temperature of liquid nitrogen (—320 F) to about 390 F under severe thermal cycling and mechanical stress and in contact with water or highly active corrosives. Other physical properties of trifluorochlorethylenes are shown in Table 1.

Chemical Resistance

One of the polymer's outstanding properties is its chemical resistance to most corrosive agents. Fuming nitric acid, hydrogen fluoride, peroxides, concentrated and dilute mineral acids, alkalies and organic solvents are a few examples. In the case of a few hydrocarbons, particularly the highly halogenated hydrocarbons, some absorption and swelling may take place but does not preclude use of the polymer with these materials. Most classes of compounds do not swell the plastic more than a few tenths of one percent at room temperature. Chief exceptions are anhydrous chlorine (12.3 percent), ethyl ether (3.8 percent) and highly chlorinated or fluorinated compounds. The polymer is subject to attack by molten alkali metals at elevated temperatures. Additional chemical resistance and dimensional stability values for trifluorochlorethylene are listed in Table 2.

Compressive Strength

Trifluorochlorethylene polymers' exceptional resistance to corrosives is one part of the unique property balance which comprises its suitability for structural use in corrosion control. The amorphous plastic has a compressive strength of 30,000 psi and good impact resistance. Its high compressive strength (for a plastic) is indicative of the material's low cold flow properties, a quality important in its use as gaskets, seals, "O" rings and packings. The term "cold flow" as applied to plastics means permanent deformation of the plastic when subjected to pressure. In this respect, the polymer deforms slightly under load but has good elastic memory.

Dielectric Properties

The polymer has excellent dielectric properties offering insulation resistance of 15,000 to 25,000 volts under thermal cycling and high humidity. Electrical properties are enhanced by the plastics zero water absorption, non-wetting and non-adhesive properties which prevent grounding due to surface water, fungus, corona formation of flash-over. Extended arc resistance, non-carbonizing at elevated temperatures and non-flammability are other dielectric characteristics.

Water Absorption

Unlike most thermoplastics, the polymer has zero water absorption, its dimensional stability being unaffected by direct contact or immersion in water. In continuous tests on moisture vapor transmission through extruded film (3 mil) over one month's time the material remained impermeable under conditions of 95 percent relative humidity at 100 F. Tests with air and a number of chemicals show that the polymer film also has low permeability to these agents.

Dispersion Coatings

The unique balance of chemical and physical properties of this polymer also applies to baked-on coatings of the polymer dispersion used as a protective lining or coating for plant equipment such as vessels, tanks, and pumps. Containing approximately 40 percent plastic in an organic thinner, these dispersions can be applied to a number of surfaces by spraying, dipping or spreading to provide optimum durability and chemical, thermal and electrical resistance. They are supplied for application in "ready-to-use" viscosities.

The usual preparatory practices for applying other organic and inorganic coatings should be followed. All surfaces to be coated must be degreased and sandblasted with coarse abrasive grit and moderately high velocity air stream to provide good tooth for the coatings. Also, the working area must be free of contamination by dusts or vapors from other coating materials in use or previously used. Even minute traces of less stable materials in a fluorocarbon film would significantly downgrade its protective properties, especially in highly corrosive service where such inclusions could create pinholes.

After application of the dispersion, the organic thinner is evaporated, leaving the white powder covering the surface. The discrete particles in sizes varying from 1 to 20 microns are fused by heating the object from 475 F to 525 F at which temperatures the white powder slowly changes to a clear transparent and continuous film. Each coating properly applied will measure 1.5 to 2.0 mils in thickness. Several coats are recommended for a standard protective film to attain a 0.010 to 0.012 mil thickness for a standard, non-permeable, protective film.

After fusion, dispersion coatings should be quick quenched in water. Films of this type, cooled slowly to room temperature, become crystalline with high tensile strength but with poor elongation and flexibility. Rapid quenching provides amorphous coatings which are transparent, tough and more extensible, flexible and impermeable to corrosives than slowly cooled coatings.

Most ferrous and non-ferrous alloys, glass, glass cloth, ceramics and plated metals can be coated. Surface irregularities must be minimal since dispersions will not fill them by capillary action but will bridge, possibly causing failure during service. Metals such as copper-base alloys with surface activity at elevated temperatures may cause peeling of coatings due to oxide formation. Copper-base alloys also can catalyze degration of the coating. Tin, nickel, silver, tin lead alloys or cadmium plating reduces this activity.

Dispersion coatings offer protection against strongly corrosive agents such as acids, alkalies and oxidants. In addition, coated surfaces, are anti-sticking, non-contaminating and physiologically inert. The one major limitation is the high (475 F to 525 F) temperature required to fuse the resin particles into a continuous film.

Plastic Laminate

Heat-sensitive materials (aircraft aluminum, wood) porous surfaces (concrete, cast iron) and large fixed tanks and vessels can be covered with a continuous layer of plastic laminate under ambient conditions.

The plastic laminate consists of a thin continuous layer of the plastic compression molded to glass cloth. The plastic extends far enough into the glass structure for a strong mechanical bond between plastic and fabric. Enough glass cloth remains exposed on the laminate's

(Continued on Page 16)

★ Revision of a paper titled "Fluorocarbon Resins for Corrosion Control" by Fred W. Troester, Minnesota Mining and Manufacturing Co., Los Angeles, Cal., presented at the 14th Annual Conference, National Association of Corrosion Engineers, San Francisco, Cal., March 17-21, 1958.

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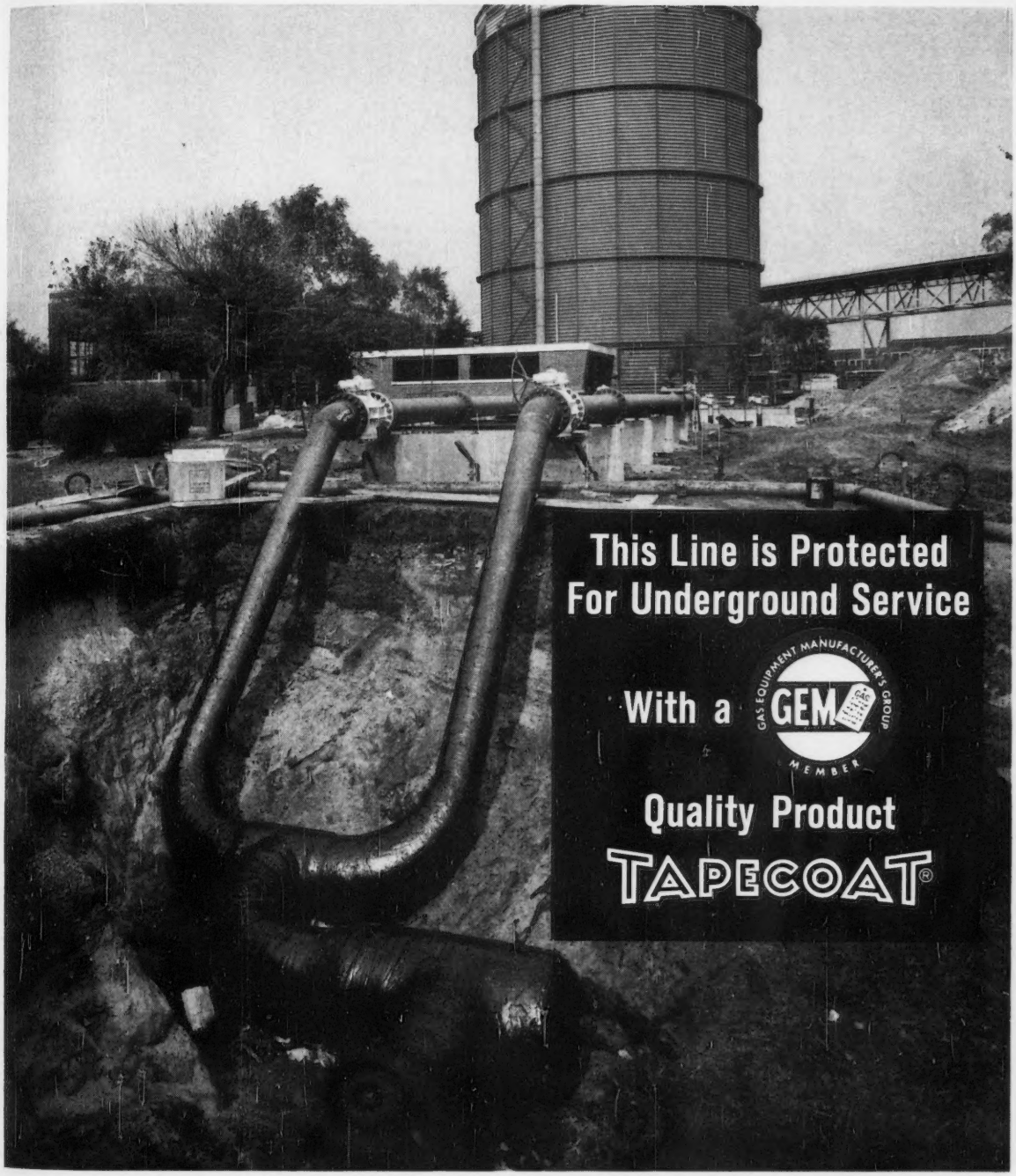

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Trifluorochlorethylene –

(Continued From Page 14)

reverse side to provide a gripping surface for conventional adhesives. In standard weight laminate, the effective thickness of the plastic above the glass fibers is 10 to 12 mils. Total thickness of the laminate is 23 to 26 mils.

Techniques for installing the plastic laminate are being improved, but a number of practical methods of lining tanks, vessels, containers and pipe involve the following basic steps.

1. Cleaning and roughening by grit blasting, sanding or etching the surface to which laminate is to be cemented.
2. Cutting and fitting laminate.
3. Apply adhesive to laminate and supporting surface, using fluid epoxy-Buna N formulations.
4. Holding laminate tightly in place while adhesive sets.
5. Sealing the seams between adjacent sheets of laminate.
6. Testing electrically for pinholes, incomplete seams.

TABLE 1—General Properties of Unplasticized Trifluorochlorethylene Polymer Grades 270-300

PROPERTIES	ASTM Test Method	Unit of Measure	Results
Impact Strength, Izod, Notched, 77 F	D256-47T	Ft lb/in of notch	3.62
Outdoor Aging, One Year	.....	.....	No detectable change
Rockwell Hardness	D785-48T	R scale	111-115
Durometer Hardness	D676-47T	D scale	80
Abrasion Resistance:			
59.9946 gm. Heat Treated	Fed. Spec. L-P-406-a method 1091	Loss in gm/1000 cycle	0.0048
57.3753 gm Quenched	Fed. Spec. L-P-406-a method 1091	Loss in gm/1000 cycle	0.0175
Shear Strength	D732-46	Psi	5,800
Thermal Conductivity	.....	Cal/cm²–sec/C/cm	6.0 x 10-4
Thermal Conductivity	.....	Btu/ft²/hr/F/in	1.74
Specific Heat	.....	Cal/gm/C	0.216
Thermal Coefficient of Linear Expansion:			
—80 to 20 C	D696-44	In/in/C	4.5×10^5
20 to 150 C	D696-44	In/in/C	7×10^5
Resistance to Heat (Continuous)	.....	F	390
Water Absorption	D570-42	Percent	0.00
Burning Rate	.....	.....	Nil
Effect of Sunlight	.....	.....	None
Effect of Strong and Weak Acids	.....	.....	(See Table 3 for complete list of chemicals)
Effect of Strong and Weak Alkalis	.....	.....	
Effect of Organic Solvents	.....	.....	Generally unaffected. Halogenated compounds cause sligh swelling.
Effect on Metal Inserts	.....	.....	Inert
Machining Qualities	.....	.....	Excellent
Clarity	.....	.....	Transparent to Translucent
Color Possibilities	.....	.....	Unlimited
Radiation Resistance	.....	.....	Embrittles at 8 Megareps
Molding Qualities	.....	.....	Excellent
Transition Temperature	.....	F	410-414
Compression Molding Temperature	.....	F	445-500
Compression Molding Pressure	.....	Psi	500-15,000
Injection Molding Temperature	.....	F	440-580
Injection Molding Pressure	.....	Psi	5,000-40,000
Compression Ratio	.....	.....	2.0
Mold Shrinkage	.....	In per in	0.005-0.010
Specific Gravity	D792-48T	.....	2.1
Apparent Bulk Factor	.....	Cu in/lb	28.8-91.0
Refractive Index	D542-42	ND^{25}	1.43
Tensile Strength, 77 F	D638-46T	Psi	4,600-5,700
248 F	D638-46T	Psi	460-570
Tensile Strength (Oriented Film and Fibre)	.....	Psi	30,000-50,000
Yield Strength 0.2% Offset, 77 F	D638-46T	Psi	3640
Elongation	D638-52T	Percent	125-175
Modulus of Elasticity, 77 F:			
Tensile	D638-46T	Psi	$192\text{-}226 \times 10^3$
Flexural	D790-45T	Psi	182×10^3
Compressive	D695-44T	Psi	$177\text{-}191 \times 10^3$
Compressive Strength, 77 F	D695-44T	Psi	32,000-80,000
Flexural Strength, 77 F	D790-45T	Psi	8200
158 F	D790-45T	Psi	1500
Stiffness, #300 NST Grade:			
Minus 297 F	D747-48T	Psi	810×10^3
Minus 148 F	D747-48T	Psi	500×10^3
32 F	D747-48T	Psi	250×10^3
212 F	D747-48T	Psi	22,000
392 F	D747-48T	Psi	4,500

Fluorocarbon Copolymer Coatings

Fluorocarbon copolymer of chlorotrifluoroethylene and vinylidene fluoride is soluble and is designed for use in air drying corrosion resistant paints and lacquers. Being generally soluble in ketones, esters, and cyclic ethers and insoluble in hydrocarbons, solutions of the resin can be made in various solvent systems. Such coatings applied by spray, brush, dip and flow techniques find applications where baking is impractical (large vessels, in place installations) or where the substrate (wood aircraft aluminum) cannot withstand the high fusion temperatures required for insoluble dispersion-type resins.

In designing a lacquer for spray applications, certain basic principles must be followed. The formulation must be balanced: that is, solvent release must proceed at a constant rate to eliminate orange peel and must be essentially complete to prevent blistering due to entrapped volatile components to which the film is not permeable. The highest practical solids content consistent with spraying viscosity should be used to realize a high build and minimize porosity.

The lacquers should be applied to degreased surfaces which are free from rust and scale. Non-adherent paint films or residual organic coatings should be removed prior to application.

The formulations suggested in Table 3 are not intended to be limiting but are simply the product of laboratory experience to date.

This lacquer is conveniently prepared by dissolving the resin in methyl isobutyl ketone to form a 45.5 percent stock solution. The required amounts of amyl acetate and light oil are then added to reduce the resin solids to 30.0 percent. Light oil is present as an anti-webbing agent and to aid in the film flow out on the coated object. The above lacquer formulation is applied by hot spray technique, the material heated to 160 F and sprayed at 20 to 40 psig pressure.

If hot spray equipment is not available or is not applicable, the same formulation may be reduced to 20.0 percent (wt.) solids with a 75/25 (wt.) mixture of methyl isobutyl ketone/amyl acetate. This lacquer is sprayed at 40 to 50 psig pressure and requires approximately six coats to build up a 10 mil film.

Applications of a lacquer by dip and flow techniques superimposes the condition of rapid set on the formulation. A more volatile solvent system is therefore required. The formulation given in Table 4 has been found suitable for this operation.

The lacquer is prepared by dissolving the resin directly in the calculated amount of solvent mixture.

Objects to be dip or flow coated should be removed from the lacquer

(Continued on Page 18)

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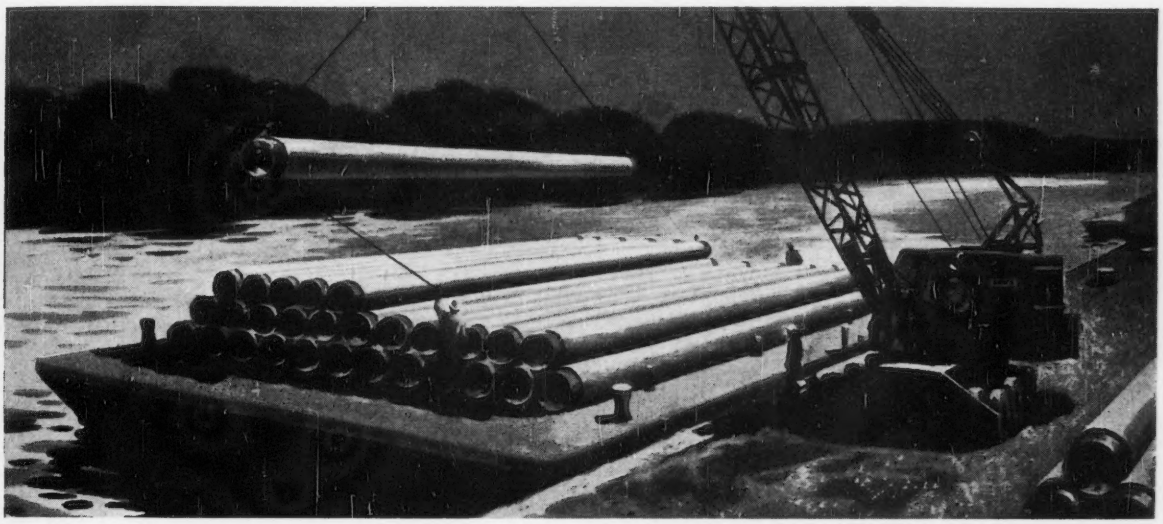

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Trifluorochlorethylene—

(Continued From Page 16)

reservoir at the rate of approximately 3-4 inches/minute to avoid sagging and building of an excessive amount of material. A minimum of 30 minutes air drying should be allowed between successive coats.

If a lower viscosity lacquer is desirable, thinning with methyl ethyl ketone is recommended.

High Molecular Weight Fluorocarbon Resins

Properties are given in Table 5 of a new fluorocarbon resin formulation which has higher molecular weight, thus making it insoluble in common lacquer solvents and giving it greater form stability at elevated temperatures.

One of the significant properties of this new resin is its excellent resistance to attack and penetration of powerful oxidants such as propellant grade red fuming nitric acid and 90 percent hydrogen peroxide. In addition, the resin does not deplete the oxidizing strength of these chemicals excessively.

The newly formulated resin may be molded, calendered or extruded into a variety of shapes. The calendering of film and the extrusion of film and tubing can be accomplished with conventional equipment, producing a material which is clear, tough and flexible. Because the polymer is amorphous, it has no sharp melting point but rather a softening range (320 to 350 F) over which the melt viscosity falls markedly. Processing temperatures in excess of the softening range are recommended (normally 375 to 450 F).

(Continued on Page 24)

TABLE 2—Chemical Resistance and Dimensional Stability of Unplasticized and Quenched Trifluorochlorethylene

Chemical	Percent Change at 25° C., 7 days			
	Sample Dimensions			Weight Change
	Thick	Width	Length	
Acetic Acid	0.16	0.05	0.00	0.0
Acetic Acid 5%	—0.16	—0.03	—0.03	0.0
Acetic anhydride	0.16	0.02	—0.03	0.0
Acetone	0.24	0.04	0.00	0.1
Acetophenone	0.00	—0.04	0.00	0.0
Acetyl chloride				0.1
Allyl Chloride				0.2
Ammonia, anhydrous				0.0
Ammonium hydroxide 10%	—0.50	—0.08	0.00	0.0
Amyl acetate	0.08	—0.04	—0.07	0.0
Amyl acid phosphate				0.0
Aniline	0.00	0.00	—0.03	0.0
Antimony pentachloride	0.00	0.02	0.00	0.0
Aqua Regia				0.0
Aroclor 1242	0.00	0.01	0.03	0.0
Aroclor 1248	0.00	0.03	0.00	0.0
Aroclor 1254	0.00	0.03	—0.03	0.0
Benzaldehyde	0.16	0.00	—0.07	0.0
Benzene	0.56	0.03	0.0	0.2
Benzonitrile				0.0
Benzyl alcohol	0.16	0.00	0.00	0.0
Benzoyl chloride				0.0
Bromine	0.00	—0.04	—0.03	0.0
Bromobenzene	0.08	0.01	0.00	0.0
Butyl acetate				0.3
Butyl alcohol	0.0	0.01	0.0	0.0
n-Butyl ether	0.17	0.04	0.00	0.0
n-Butyl sebacate	—0.08	0.00	0.00	0.0
Calcium chloride (sat)				0.0
Carbitol acetate				0.0
Carbon disulfide	0.16	—0.02	—0.03	0.1
Carbon tetrachloride	0.5	0.0	0.1	0.4
Cellosolve acetate				0.0
Chlorine anhydrous (147 hrs.)	6.6	5.0	4.8	12.3
1-Chlor.-1 -nitropropane	—0.16	—0.02	—0.03	0.0
2-Chloropropane				0.3
o-Chlorotoluene				0.0
p-Chlorotoluene				0.0
Chlorotrifluoroethylene (8° to 25° C.)	9.5	2.2	1.0	9.1
Chlorotrifluorethylene liq. polymer				0.0
Chromic acid cleaning solution				0.0
Cresol	—0.08	—0.01	0.0	0.0
Cyclohexanone				0.0
Dibutyl phthalate	0.0	0.0	0.03	0.0
Dibutyl sebacate				0.0
1, 2-Dichlorobutane				0.0
3-Dichlorethyl ether	0.17	0.08	0.0	0.0
1, 2-Dichlorohexa-fluorocyclobutane				0.1
Dichloropropylene	0.16	0.01	0.0	0.0
2, 4-Dichlorotoluene				0.0
3, 4-Dichlorotoluene				0.0
Dicyclopentadiene	0.0	—0.01	0.03	0.0
Diethylamine	4.5	0.4	0.1	1.9
Diethyl carbitol	0.16	0.04	0.0	0.14
Diethyl Cellosolve	1.7	0.1	0.0	0.8
Diethylenetriamine				0.0
Dioxane	0.08	0.03	0.0	0.0
Ethanol 50%	—0.16	—0.04	—0.01	0.0
Ethanol 95%	—0.08	—0.03	—0.03	0.0
Ethyl acetate	2.3	0.2	0.1	1.2
Ethyl n-butyrate	0.9	0.1	0.0	0.5
Ethyl ether	8.0	1.2	0.5	3.8
Ethyl formate				0.2
Ethyl propionate	1.8	0.1	0.1	1.0
Ethylene chloride				0.0
Freon 113	1.2	0.1	0.0	1.2
Formic Acid				0.0
Furan	4.1	0.4	0.1	2.4
Halowax 1000	0.0	—0.02	0.0	0.0
n-Heptane	—0.08	—0.07	—0.07	0.0
Hollingshead H-2 (7 days)				
Hydrochloric acid 10%	—0.16	—0.05	—0.07	0.0
Hydrofluoric acid (anhy.)				0.0
Hydrofluoric acid 50%	0.08	—0.09	—0.07	0.0
Hydrogen peroxide 3%	0.0	—0.01	—0.03	0.0
Hydrogen peroxide 30%				0.0
Isopropyl ether				0.2
Methanol	0.08	0.02	—0.03	0.0
Methyl acetate	1.8	0.1	0.0	1.0
Methylal	2.5	0.4	0.1	1.3
Methallyl chloride	0.25	0.06	0.10	0.1
Methyl n-butyrate	1.6	0.2	0.0	0.8
Methylene chloride				0.2
Methyl ether	6.9	5.4	4.9	6.4
Methyl ethyl ketone				0.2
Methyl formate				0.1
Methyl propionate	2.8	0.1	0.0	1.4
Mineral Oil	0.08	0.02	0.0	0.0
Naphtha Solvent	0.08	0.01	—0.03	0.0
Nitric Acid	0.16	0.0	0.0	0.0
Nitric Acid fuming 89–95%				0.0
Nitric Acid 10%	—0.24	—0.08	—0.03	0.0
Nitrobenzene	0.0	0.01	—0.03	0.0
Nitromethane	0.0	—0.02	—0.07	0.0
Oleic Acid	—0.08	—0.07	—0.07	0.0
Pentachloroethane				0.0
Perchloric Acid (14 days)	0.0	0.0	0.0	—0.2
Perfluorotriethylamine				0.0
Piperdine				0.0
Potassium hydroxide 10%	0.0	0.0	0.0	—0.2
Propylene chloride				0.0
n-Propyl acetate	1.1	0.2	0.1	0.6
n-Propyl ether				0.3
n-Propyl formate				0.1
n-Propyl propionate	0.6	0.2	0.0	0.4
Pyridine	0.08	0.05	—0.03	0.0
Santicizer B-16				0.0
Santicizer E-15				0.0
Santicizer M-17				0.0
Santicizer 8				0.0
Santolube 31				0.0
Sodium carbonate 2%	—0.16	—0.11	—0.03	0.0
Sodium chloride 10%	—0.08	—0.08	—0.03	0.0
Sodium hydroxide 1%	—0.16	—0.10	—0.03	0.0
Sodium hydroxide 10%	—0.16	—0.14	—0.03	0.0
Stannic chloride	0.08	—0.02	0.0	0.0
Sulfuric acid	0.16	0.0	—0.03	0.0
Sulfuric acid 3%	—0.40	—0.05	—0.07	0.0
Sulfuric acid 30%	—0.08	—0.04	—0.07	0.0
Sulfuric acid 20% free SO_3				0.0
Sulfur dioxide, anhydrous				0.1
sym-Tetrachloroethane	—0.24	—0.05	0.0	0.0
Tetrachloroethylene	0.8	0.0	—0.1	0.8
Toluene	0.9	0.1	0.1	0.4
1, 1, 1-Trichloroethane				0.1
1, 1, 2-Trichloroethane	0.08	0.01	—0.03	0.0
Trichloroethylene	2.9	0.2	0.1	2.3
1, 2, 3-Trichloropropane				0.0
Trichloropropane	—0.24	0.0	0.0	0.0
Triethylamine				0.2
m Xylene	1.0	0.1	0.0	0.4

NOTE: Changes in weight do not indicate chemical reaction but rather absorption. Furthermore, through development of crystallinity by heat treatment it is possible to reduce absorption to a minimum.

TABLE 3—Lacquer Formulation No. 1

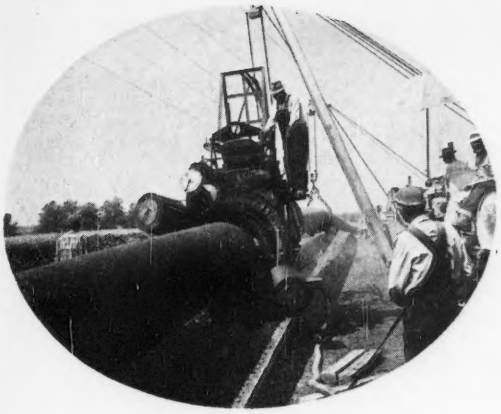
Non-Volatile Composition	
Fluorocarbon copolymer	100.0% (wt)
Volatile Composition	
Methyl isobutyl ketone	51.3% (wt)
Amyl acetate	46.6
Light oil	2.1
Physical Constants	
Solids, % weight	30.0
Viscosity cp., at 77 F	800
at 160 F	126
Density, lbs per gal	8.2
Coverage, sq ft/gal/mil thickness	226

TABLE 4—Lacquer Formulation No. 2

Non-Volatile Composition	
Fluorocarbon copolymer	100.0% (wt)
Volatile Composition	
Methyl ethyl ketone	45.3% (wt)
Methyl isobutyl ketone	57.7
	100.0% (wt)
Physical Constants	
Solids % (wt)	30.0
Viscosity cp., at 77 F	2420

TABLE 5—Properties of High Molecular Weight Fluorocarbon Resins

PROPERTY	Test Method	Nominal Value
Physical form		Free flowing, white powder
Color and Clarity	1/16-inch molded sheet	Transparent, colorless
Solution Viscosity, cs.	JCT-7	1.5
Softening Temp, F		320
Compression Molding Temp, F		450
Specific Gravity, 75 F	Jolly balance	2.02
Refractive Index, N_D, 85 F	ASTM D-542-50	1.416
Tensile Strength, 77 F, psi	ASTM D-638-52T Die II, 0.2 in./min	3400
Yield Point, 77F, psi	Die II, 0.2 in./min	1020
Elongation, 77 F, percent	Die II, 0.2 in./min	230
Hardness, Shore D, 77 F, 5 sec	ASTM D-676-49T	64
Impact Strength, Izod, Notched 77 F	ASTM D-256-47T	No break, 20 lb hammer
Water Absorption, Percent	ASTM D-570-42	<0.01
Flammability		Non-flammable
Acid Resistance (including fuming nitric)	Immersion and "H" cell	Excellent
Hydrocarbon Fuel Resistance	Immersion	Very good
Solvents		Cyclic ethers



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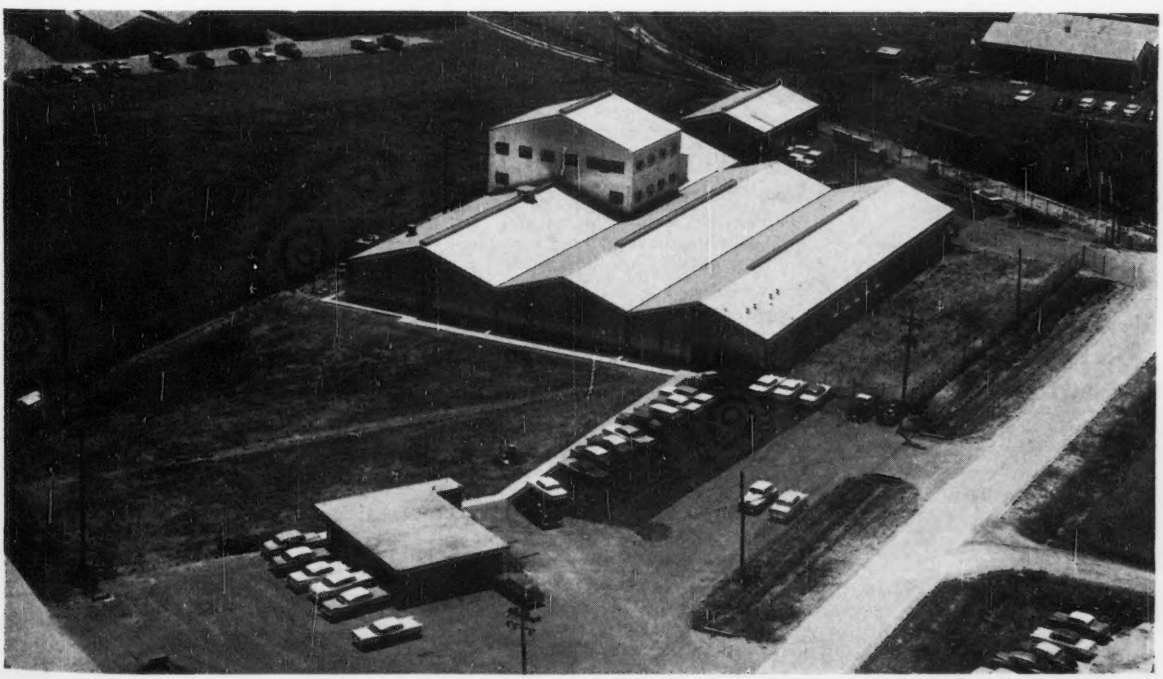

Engineering Approach to a CHEMICAL PLANT COATING PROGRAM*

(Parts 1 and 2 were published in the January issue)

Part 3—Some Coatings Systems and What Was Learned About Them

Abstract

Specifications of some of the coatings systems used at a seacoast chemical plant are given. Some results accomplished with these coatings in the environment are described. Coatings include vinyls, epoxies, oil base, materials, zinc-silicones, aluminum metallizing. 5.4.6

IN ORIGINAL design of and later in planning new construction of the Union Carbide Chemicals Company's Seadrift, Texas plant, specifications for several corrosion control systems were prepared. Among these the protective coating systems were planned to give long time protection to all exposed metal surfaces and at the same time to evaluate different materials by field test.

Specifications called for nearly all surfaces to be 100 percent sand blasted. A schedule of these specifications is given in Table 1.

TABLE 1—Coating Specifications Schedule

Exterior Specifications

1. Structural Steel, Piping and Tanks
   - A. 1 Coat Modified Vinyl Primer
     2 Coats Vinyl
     4½ Mils Minimum Thickness
   - B. 1 Coat Modified Vinyl Primer
     2 Coats Vinyl Hot Sprayed
     6 Mils Minimum Thickness
   - C. 1 Coat Zinc Silicate Formulation
     1 Coat Modified Vinyl Primer
     1 Coat Vinyl Hot Sprayed
     6 Mils Minimum Thickness
2. Tanks White
   - A. 1 Coat Vinyl Wash Primer
     1 Coat Vinyl Intermediate
     1 Coat Vinyl Grey
     1 Coat Vinyl White
     4½ Mils Minimum Thickness
   - B. 1 Coat Modified Vinyl Primer
     1 Coat Vinyl Hot Sprayed
     2 Coats Vinyl White
     6 Mils Minimum Thickness
3. Tanks Special Test
   - A. 1 Coat Epoxy Ester Primer
     2 Coats Epox Ester White
     4½ Mils Minimum Thickness
   - B. 1 Coat Vinyl Wash Primer
     2 Coats Epoxy Ester White
     4½ Mils Minimum Thickness
   - C. Pickling and Phosphate Treatment for Steel Not Blasted
     1 Coat Oil Base Primer
     2 Coats Alkyd Resin Formulations
     4½ Mils Minimum Thickness

Interior Specifications

4. Structural Steel, Piping and Tanks
   - A. Prime Coat Oil Base
     Finish Coat Oil Base

Hot Surfaces Specifications

5. Temperatures 140F to 1000F
   - A. One Coat Zinc Silicone Formulation
6. Temperatures 140F to 500F
   - A. 2½ Mil maximum thickness zinc silicate formulation sprayed on cold surface and treated or sprayed directly on hot surfaces with treatment not necessary.
7. Temperatures to 1400F
   - A. 3 to 5 mils hot sprayed aluminum metallizing
     Stack to be coated should be preheated to 150F-1180F
     Sprayed with aluminum metallizing wire
     Coated with silicate of soda
     Baked at 400F for four hours

★ Presented under the title "Field Experience With Corrosion Resistant Coatings," by Newell D. Casdorph, Union Carbide Chemicals Corp., Port Lavaca, Texas, at the 15th Annual Conference of the National Association of Corrosion Engineers, Chicago, Ill., March 16-20, 1959.

While the protective coating exposure of the Seadrift Plant is considered mild from a chemical manufacturing company standpoint, a major factor in the corrosion control problem is its location in the hot, humid Gulf Coast area.

Some Experience with Vinyls

Statements concerning performance of generic materials in this article are based on observation made on a limited number of manufacturers products. Formulation of these materials by other companies may show entirely different corrosion resistant qualities.

Careful application of the 1A Vinyl System gives good corrosion protection. It is difficult for some applicators to build the materials up to 4½ mils minimum thickness with three coats cold sprayed and the irregular surfaces which need the most protection usually are not well covered.

When the coating is applied less than the specified mil thickness, rust will appear on flat surfaces and sharp edges in a few month's time.

The 1B Vinyl System is similar to 1A except the vinyl coats are hot sprayed, bringing the total thickness to a minimum of 6 mils. Hot spraying tends to build up over rough surfaces and corners. The average spray man can make this application easily so when this system is used corrosion problems for several years should be limited to touching up mechanical damage, field welds which were not properly cleaned, etc.

A zinc silicate formulation on the sandblasted steel (System 1C) with vinyl overcoating offers the cheapest cost per square foot per year approach to a long range protective coating system. Mechanical damage to the coatings is of little concern because the zinc prevents the steel from rusting. Follow up cost should be limited for several years to touching up field welds which may have not been properly prepared for coating and spot repairs to the vinyl coatings which have been mechanically damaged.

White Vinyl System 2A gives good protection of flat surfaces. The remarks under 1A concerning irregular surfaces apply to this system.

Remarks under 1B generally apply to the 2B system. One color vinyl coat is hot sprayed in order to build up the mil thickness, get good sharp edge build-up and the two final white coats are cold sprayed to get good coverage.

Uses for Epoxy Esters

3A and 3B Epoxy Ester system coated tanks were very well protected on the flat surfaces. As with other systems, trouble occured on irregular surfaces in a few months' time. Compared to other systems considerable chalking occured. Some difficulty was experienced in making spot repairs due to the material flaking off. Epoxy materials are being used to an advantage where resistance to solvents is needed.

Alkyd Coating System 3C after three years' exposure to the weather is giving good service. The side exposed to prevailing winds contaminated by alkalies from the water cooling tower is now beginning to fail.

Oil Base 4A System gives very good service on interior surfaces free from moisture. This coating deteriorates rapidly in the Gulf Coast area climate when used below floor levels, under roof overhang and locations which are exposed to moisture.

Zinc Silicone Formulation System 5A has limited life expectancy in the 140—400F range at the Seadrift Plant.

Zinc Silicate System 6A appears to have long life expectancy in the lower temperature range and after two years' exposure in 400F service seems very promising.

System 7A in service at temperature ranging up to 1400F has proved satisfactory. No flaking off of coating has occurred. Some discoloration caused by rusting due to pin holes or porosity has taken place. This is being corrected by light brush blasting and overcoating with a high temperature aluminum silicone formulation seal coat.

Part 4—Investigating Sandblasting and Coating Application Hazards

Abstract

Tests are described in which an effort was made to ignite gases from solvents commonly found in coatings. In no case was it possible to ignite the solvents with the sand particles. Sandblast streams played on burning solvents extinguished them. Means are described to prevent sandblasting from causing damage to adjacent operations and to reduce objections by operating personnel to sandblasting. 5.9.3

EXTENSIVE EXPERIMENTATION has been carried out using several highly inflammable liquids and/or gases trying to ignite them by sparks produced by sandblasting. An iron table (Figure 1) with high backstop, forced liquid material feed bottles (Figure 2) and conventional sandblasting equipment were used in carrying out these tests.

(Continued on Page 22)

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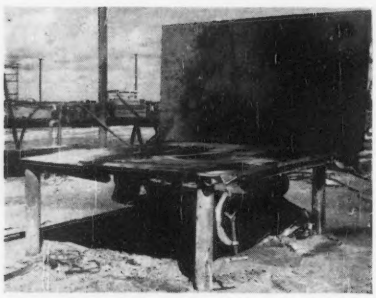

Figure 1—Iron table with backstop, forced liquid material feed bottles and conventional sandblasting equipment used in tests.

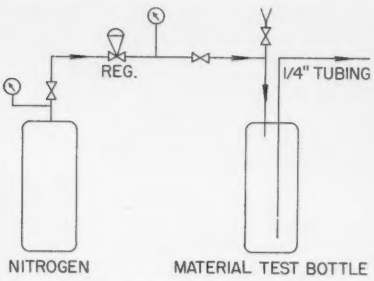

Figure 2—Diagram of forced material feed for sandblasting hazards investigation.

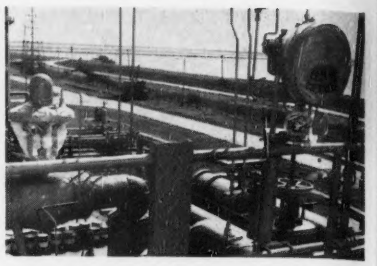

Figure 3—Transparent plastic film used to protect instruments, pneumatic motor valves and other equipment from sandblasting dust.

Engineering Approach –

(Continued From Page 20)

Table 1 shows several common materials some of which were used in the testing program.

Acetaldehyde, kerosene and gasoline include in a representative way extremes of low ignition temperatures, low flash points, low boiling points, low explosive limits and wide explosive ranges. The series of tests listed below were carried out with these three materials. (Note: table top and back plate were preheated to vaporize kerosene.)

With the sandblasting operation going on, liquids were fed into the air stream from the sandblast nozzle to the surface of the steel being blasted and to the table top under the blast area. Blasting was carried out at right angles to the back plate, at a 45 degree angle slanting up and rotated thru a 180 degree arc to a slanting down position. Materials used in these tests were never ignited by sandblasting.

The next step in the test program was to wet the back plate and the table top with liquid, ignite and keep squirting material into the blaze until it was burning vigorously and then turn the sandblast unit on while still adding fuel. The blasting operation rapidly extinguished the fire while the liquid was still evaporating and/or boiling. No re-ignition occured with any of the materials used.

The large volume of expanding air from the nozzle makes it extremely unlikely an explosive mixture concentration will be reached in the sand blasting area. This would indicate there is little danger in working where there may be small gas leaks present.

The particles material made incandescent by friction (not hot enough to be seen in daylight) are very small and are carried by the expanding air flow until at the time they reach the surrounding atmosphere their temperature has dropped below the point at which they will ignite any material.

The experiment mentioned above, blasting with the nozzle slanted up with evaporating or boiling materials a few inches below further indicate the hot particles are light enough to be carried by the air flow. Any large particles of rust or scale which may be heavy enough to fall out of the air blast stream have so much volume the energy in the blast sand particles is not great enough to heat them to a dangerous temperature.

However, it should be emphasized that sandblasting should never be carried out where flammable liquids are spilled or leaking or large gas leaks are present.

A large semi-portable fan should be used to change the air where gas concentrations may occur. The area then should be checked with a flammable material gas analyzer prior to sand blasting.

Spray Painting Fire Hazards

Most corrosion resistant coating formulations will burn under proper conditions. Table 1 gave the flash and ignition point of representative materials. No solvents or coatings have lower flash, boiling or ignition points than materials listed or tested.

Ignition and flash-back tests were carried out by spraying coating materials and solvents commonly used directly into a hot controlled kerosene fire. Regardless of material used no flash back or burning of sprayed mixture occured less than 24 inches from the spray gun head.

The only time the flame approached the nozzle was when the material was sprayed on the surface between the burning kerosene and the spray nozzle. As the solvent evaporated flash-back would occur, although at no time did the operator seem to be in danger.

Only a very limited hazard exists when spraying near furnaces or hot lines provided simple precautions are taken. For example, in open or outside areas the operator should always spray downwind. Closed buildings or places where very little air movement is present should be ventilated or the air should be changed by portable fans to prevent possible explosive mixtures of solvents and air from occuring.

The spray nozzle should be grounded to prevent static electrical discharges from the spray head to the steel being coated.

Equipment Damage From Sand Blasting

The human element, aggravated by the nuisance value of having sand flying around and underfoot causes resistance to sandblasting out of proportion to actual damage it does. A realistic approach must be made to the operating people responsible for an area in which work is to be done. They must be sold on the idea an all-out effort will be made to keep sand or dust from settling on any type of equipment which may be damaged or have its operation affected.

In doing touch up work sandblasting should be held to the minimum amount necessary to get good surface preparation. The amount of sand used can be reduced by equipping all blasting units with fast shut off type remote controls. To further reduce use of sand, vacuum feed type blasting guns should be used for all light work.

Each day after blasting is finished all sand should be cleaned up and hauled away. Good housekeeping will reduce the nuisance and resistance to the work will be much less.

Instrument tubing and insulation adjacent to spots to be blasted can be protected by using old rubber inner tubes. Transparent plastic film (Figure 3) is ideal for sealing instruments, pneumatic motor valves and other equipment from sand or dust.

Work should be planned to take advantage of wind direction. By careful timing work can be done on high columns or structures with the sand and dust carried away by the wind where no damage will be done. Polyester film shrouds will cause the sand to drop straight down and prevent it spreading it over large areas.

Motor driven centrifugal pumps and other rotating equipment can be adequately protected by covering them with sisal kraft weather resistant paper.

Under some conditions large semi-portable fans can be used to keep sand or dust away from equipment. Again ad-

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TABLE 1—Characteristics of Flammable Materials Used in Painting

No.	Material	Degrees			Explosive Limits % By Volume	
		Flash Point	Ign. Temp.	Boil Point	Lower	Upper
1.	Ethyl Ether	—49	356	95	1.9	48.0
2.	Acetaldehyde	—36	365	70	4.1	55.0
3.	Kerosene	100	490	304/574	0.7	5.0
4.	Gasolene	—45	536/800	100/400	1.4	7.6
5.	Ethylene Oxide	0	804	51	3.0	80.0
6.	Methyl Ethyl Keytone	30	960	176	1.81	10.0
7.	Acetone	0	1000	134	2.6	12.0

CYANAMID

CORROSIONEERING WITH LAMINAC®

POLYESTER RESINS

Two years in formaldehyde—reinforced Laminac lining surpasses its own record

This test fully confirms the faith LAMINAC inspired 31 months ago when this 200,000-gallon tank was coated with Cyanamid's polyester resin. Since then the tank has held a 37% solution of formaldehyde at 90°F. without corrosion even at the unprotected ceiling line.

The lining is fabricated of glass cloth and LAMINAC Polyester Resin 4109, with a clear finish coat of LAMINAC Resin 4111. It was installed on May 20, 1957, replacing a vinyl-based lining, the service life of which had been shortened to a point where the underlying concrete was deteriorating, and contaminating and discoloring the formaldehyde.

The results are also in on another stiff test given to LAMINAC! Starting Dec. 20, 1958, 200,000 gallons of a 44% solution of formaldehyde were placed in a tank at 120°F. In addition, the ceiling was coated to prevent corrosive effects. The solution remained in the tank for 1 year.

At the end of 1959, inspection revealed no breakdown or blistering, pitting, bleaching, cracking or pulling away from the LAMINAC-lined concrete wall!

The reinforced LAMINAC linings for these huge formaldehyde tanks are expected to last indefinitely.

If you have a tough corrosion problem, reinforced LAMINAC may well be the answer. For details or technical assistance, get in touch with any Cyanamid office listed below.

CYANAMID AMERICAN CYANAMID COMPANY • PLASTICS AND RESINS DIVISION

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Trifluorochlorethylene—

(Continued From Page 18)

Fluorocarbon Film Applications

Fluorocarbon films can be readily heat sealed by thermal pulse or radio frequency techniques.

Some possible applications of these resins include film for specialized packaging, fuel cells (especially for oxidants), expellent bladders, hose liners (especially for strong acids and oxidants) and optically clear face shields, sight glasses, etc., where exposure to strong acids and oxidants is required.

Fluorocarbon Grease

Fluorocarbon grease is the product of several years' research directed toward formulation of a true polychlorotrifluoroethylene grease with outstanding lubricity, chemical resistance and thermal stability. This material has shown good resistance to attack by the reagents listed in Table 6.

This grease has been used successfully as a lubricant for glass joints or stopcocks at temperatures from 150 to 170 C (300 to 340 F), and as a lubricant and sealent of bearings, valves, packings and shafts in equipment handling halogens, corrosive chemicals or strong acids.

The fluorocarbon products discussed possess excellent chemical inertness, dimensional stability over a wide temperature range and good mechanical properties. They have proven excellent and highly valuable to the chemical industry for coatings, lining, gaskets and packings.

TABLE 6—Reagents Resisted by Fluorocarbon Grease

Oxidizing Agents	Halogens
Fuming Nitric Acid Hydrogen Peroxide Oxygen Chromyl Chloride Chromyl Nitrate Potassium Permanganate	Fluorine Chlorine Bromine Iodine Monochloride Bromine Trifluoride
Strong Acids and Corrosive Chemicals	**Miscellaneous Chemicals & Solvents**
Concentrated Sulfuric Acid Hydrogen Fluoride Hydrogen Chloride Chlorosulfonic Acid Antimony Trichloride Phosphorous Oxychloride Aluminum Chloride Trifluoromethyl Hypofluorite Nitrogen Oryfluoride Nitroxyl Fluoride	Aqueous Acids and Bases Alcohol Ether Hydrocarbon Solvents Chlorinated Hydrocarbons Fatty Acids Silanes Furanes

Technical Topics Scheduled for March

Oil-Soluble Inhibitors for Controlling Corrosion in Tankers and Pipe Lines, by W. S. Quimby

Use of Polyester and Epoxy Resins for Chemical Plant Repairs, by Walter A. Szymanski

Accelerated Corrosion in Aluminum-Silver Alloys, by H. H. Stadelmaier and E. M. Whitener

Relationship of Microstructure and Stress Corrosion Cracking of Type 410 Stainless Steel, by P. S. Trozzo and R. F. McCartney

Rubber and Synthetic Linings Give Good Sea Water Service*

RUBBER AND synthetic rubber linings have been in sea water service for 10 years with no visible change in the compound. Synthetic fuel oil delivery hose normally lasts eight years or more. Generally, the hose must be replaced because of external abrasion rather than breaking down of the rubber. Hard rubber pipe and fittings subjected to salt water and underground use have been in service for over 50 years.

If rubber lining for steel is damaged or torn in service, the lining usually can be patched by the applicator and quickly returned to service.

The price of lining, including steel preparations, is approximately $2.50 to $4 per square foot, based on flat surfaces. The price would be higher for more complicated shapes and tanks which would include a large number of steel bars, channels, supports, etc.

In marine applications where salt water corrosion is a primary concern, the cost of lined steel and/or plastic pipe and linings will be higher in installation cost than heavy gauge galvanized steel. Properly used, however, they are almost certain to give longer trouble free life than steel pipe where corrosion is a factor.

DISCUSSION

Questions by A. V. Morrison, California Oil Company, Perth Amboy, New Jersey:

1. What has been your experience in coating pump impellers?

2. Would you favor a relatively hard coating such as epoxy or a relatively soft coating such as plastisol or rubber?

3. A number of our vendors favor natural rubber rather than synthetic for sea water applications. Any comment?

Reply by J. A. Thompsen:

1. Generally speaking, we mold hard rubber coating over a metal face on the impeller shaft end. This has been satisfactory and in use for 15 or 20 years in pumps ranging from ½ to 10 horsepower at speeds from 750 to 3400 rpm. On some applications where abrasion is involved, soft rubber had been used satisfactorily, but this is usually only at 1750 rpm speeds.

2. We believe that a hard rubber coating is the best all-around preparation. At high speed operation with abrasive conditions, we have coated hard rubber impellers and volute cases with epoxy or soft rubber lining in the relatively few applications over a period of about two years. These appear satisfactorily, but we do not have long term results. We do not use plastisol coating for impellers based on problems of absorption and porosity.

3. We definitely favor natural rubber over synthetic rubber for sea water applications for reasons of moisture absorption. Natural rubber compounds are usually considerably lower in moisture absorption than synthetic compounds, so that in a molded impeller the product is superior.

In the case of hard rubber compound impellers, we would favor natural rubber over synthetic rubber because of lower moisture absorption and better adhesion of steel.

★ Revision of a paper titled "Controlling Marine Corrosion With Rubber and Plastics" by J. A. Thompsen, American Hard Rubber Co., New York City, presented at the 15th Annual Conference, National Association of Corrosion Engineers, March 16-20, 1959, Chicago, Ill.

Engineering Approach—

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vantage should be taken of the wind direction in conjunction with fans.

Figure 4 shows a 3 hp motor located in the sand blasting yard, connected to a small gear reducer drive unit by a falk coupling. Eccentric drive on the gear unit is connected to a reciprocating pump shaft type rod by a connecting rod. The shaft is sealed on one end by using seal type packing and the opposite end is running through a brass sleeve with center section of sleeve assembly being used as an oil reservoir.

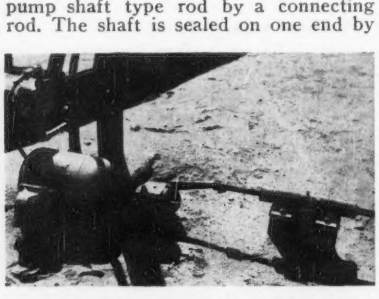


Figure 4—Electric motor and drive assembly exposed to sandblasting dust for several weeks indicate that sandblasting residues are relatively harmless to rotating equipment.

Sand shown around this installation accumulated in one week's time. The motor was run continuously and no effort was made to protect the assembly from the sand.

An installation like this can be used very effectively when interested persons are invited to inspect it. They will be amazed by what they see. Motor, Coupling and gear box are running along smoothly after weeks of exposure with no apparent damage. The shaft will have a highly polished appearance with very little evidence of wear.

The motor, coupling and gear box can be protected adequately by placing something over the top and keeping it in place. The eccentric connecting rod and shaft should be completely enclosed to keep them free of dust.

References

1. Gasoline Resistant Tank Coatings. By W. W. Crammer., CORROSION, Vol. 8, 195-204 (1952) June.

An Investigation of

Non-Metallic Pipe for Use With Liquid Corrosives*

Abstract

Metallic and non-metallic materials used for piping are discussed, showing the number of different corrosive solutions each material can handle, and comparative costs of the two groups of materials. Also discusses proper selection of non-metallics for use with liquid corrosives. Includes tabular data from investigation. 7.2

Introduction

THIS ARTICLE discusses those general areas of liquid corrosive services where non-metallic pipe can be used to best advantage. Materials considered are listed with trade names in Table 1.

A group of metallics also was selected for comparison and to establish limits of non-metallic pipe use. The materials considered were aluminum, cast iron, copper, high silicon irons, chemical lead, mild steel, Hastelloy "C", Monel and stainless steels types 304, 316, 321 and 347.

Some Comparisons of Metallics and Non-Metallics

The division of chemical piping into "metallic" and "non-metallic" is intended to emphasize the differences in physical and chemical properties. Generally, the metals have higher tensile and impact strengths, heat distortion temperatures and compressive strengths. For comparison, therefore, discussion is limited to temperatures and pressures that can be handled by non-metallics.

Chemical Resistance

Technical bulletins and corrosion resistance charts were gathered from manufacturers of the 24 materials under consideration. For each of these materials, a corrosion resistance table was prepared containing basic information on suitability of each material for each service. This table comprised a total of 289 different solutions (and concentrations of solutions), for which manufacturers made claims or indicated that their product was unsuitable. Included in this group of solutions was a set of 58 corrosive services detailed in the Chemical Engineers Handbook.[1]

Table 2 shows the results of this investigation, totaling the numbers of service conditions rated as suitable (S) or unsuitable (U) by the manufacturer or Chemical Engineers Handbook. The questionable (?) column indicates conflicting reports. With glass and Teflon, only the U column shows values. Of 58 corrosive services detailed in the numbers tabulated for each material varies from 65 (aluminum) to 160 (Saran). The value of Table 2 for purposes of direct comparison, therefore, could be limited to the numbers in the U column only. This would give a negative rather than a positive index of comparison.

Of the 24 piping materials considered, only 19 were included in the Chemical Engineers Handbook tables. Therefore, this set of 58 liquids could not be used for direct comparison either. In the original group of 289 corrosives, however, there were 22 liquids for which all materials could be rated. In this instance direct comparison can be made, and Figure 1 is a reproduction of this comparative corrosion resistance chart. Table 3 totals the satisfactory, unsatisfactory and questionable corrosion resistant ratings of the 24 piping materials, as shown in Figure 1.

Some organizations have issued reports that compare corrosion resistance by chemical groups rather than individual reagents. One such report is abstracted here.[2] Four plastics and four metals are rated for their resistance to 13 groups of chemicals. Figure 2 is a reproduction of this abstract; Table 4 totals the good, fair, and unsatisfactory ratings of Figure 2.

An expansion of this theme is a rating of materials by R. B. Seymour and R. H. Steiner.[3] Parts of their work are re-stated as Table 5. Quoted below are introductory statements by Seymour & Steiner to explain use of the table:

". . . many materials of construction have been rated on the basis of practical consideration and chemical resistance. Since the ten items under consideration are rated from 1 to 10, the perfect material would obviously have a total rating of 100. While the perfect material does not exist, these data may be used to help select materials based on the highest rating for the type of service intended."

From these data, there seems to be a broad range of corrosion resistance among the non-metallics. For example, Table 2, of improbable value for direct comparison purposes, attains significance when the materials are considered solely from the standpoint of the number of solutions they can handle. Derived from a total of the manufacturer's recommendations, the metals have ratings of 642 satisfactory and 300 unsatisfactory; the non-metals have ratings of 817 satisfactory and 163 unsatisfactory. From almost any point of view, including consideration of questionable results, these figures would be indications of a greater range of corrosion resistance among the non-metallic piping materials. This is reinforced by considering that only the unsatisfactory ratings were tabualted for glass and polytetrafluorethylene.

(Continued on Page 26)

★ Revision of a paper titled "Non-Metallic Pipe for Liquid Corrosives" by C. A. Burt, American Hard Rubber Co., Chicago, Ill., presented at the 15th Annual Conference, National Association of Corrosion Engineers, March 16-20, 1959, Chicago, Ill.

TABLE 1—Non-Metallic Pipe Materials Considered

Polyethylene	Ace Parian
Polystyrene Copolymer	Ace-Ite
Polyvinyl Chloride	Ace Riviclor
Vinylidene Chloride	Ace Saran
A Modified Phenolic, phenol-formaldehyde, furfuryl alcohol or furane resin with acid digested asbestos fiber	Haveg
Polytetrafluorethylene	Teflon
Buna-N hard rubber	Ace Tempron

TABLE 2—Totals of Reported Chemical Resistances of Pipes to 289 Service Conditions*

Piping Material	S	U	?	Totals
Polystyrene copolymer	76	25	0	101
Hard rubber	88	13	1	102
Polyethylene	65	14	1	80
Polyvinyl chloride	88	8	1	97
Vinylidene chloride	148	10	2	160
Buna-N hard rubber	70	10	0	80
Aluminum	34	26	5	65
Butyrate	42	42	1	85
Cast Iron	28	35	3	66
Copper	90	39	3	132
Glass	...	11	..	...
Hastelloy "C"	127	3	0	130
Phenolic resins	87	10	0	97
High Silicon Irons	79	11	0	90
Impervious Graphite	89	12	0	101
Chemical Lead	49	35	7	91
Monel	43	24	0	67
Stoneware	65	8	0	73
Steel (Mild)	26	40	1	47
Stainless, Type 304	40	22	4	66
Stainless, Type 316	44	21	1	66
Stainless, Type 321	41	22	3	66
Stainless, Type 347	41	22	3	66
Teflon	...	0	..	...

* As rated by manufacturers.
S = Suitable
U = Unsuitable.
? = Questionable (conflicting reports).

TABLE 3—Reported Chemical Resistances of Pipes to 22 Specific Service Conditions* (See Figure 1.)

Piping Material	S	U	?
Polystyrene copolymer	16	6	0
Hard rubber	19	3	0
Polyethylene	17	5	0
Polyvinyl chloride	22	0	0
Vinylidene chloride	18	4	0
Buna-N hard rubber	21	1	0
Aluminum	11	10	1
Butyrate	11	11	0
Cast Iron	9	12	1
Copper	15	6	1
Glass	21	1	0
Hastelloy "C"	21	1	0
Haveg	19	3	0
High Silicon Irons	17	5	0
Impervious Graphite	21	1	0
Chemical Lead	10	12	0
Monel	16	6	0
Stoneware	20	2	0
Steel (Mild)	8	14	0
Stainless, Type 304	12	9	1
Stainless, Type 316	13	8	1
Stainless, Type 321	12	9	1
Stainless, Type 347	12	9	1
Teflon	22	0	0

* As rated by manufacturers.
S = Suitable.
U = Unsuitable.
? = Questionable (conflicting reports).

TABLE 4—Summary of Corrosion Resistance to 13 Groups of Chemicals (See Figure 1.)

Piping Material	Good	Fair	Unsatisfactory
Polyethylene	8	1	4
Polystyrene Copolymer	8	3	2
Polyvinyl Chloride	10	1	2
Vinylidene Chloride	10	3	0
Carbon Steel	5	5	3
Stainless Steel (18-8)	11	2	0
Cast Iron	10	2	1
Copper	6	6	1
Totals Non-Metallics	36	8	8
Totals Metallics	32	15	5

Non-Metallic Pipe—

(Continued from Page 25)

Further substantiation is derived from Table 3, where direct comparison was made of the corrosion resistance to 22 specific liquids. Totals for metal pipe were 156 satisfactory against 101 unsatisfactory. Totals for non-metallic pipe were 227 satisfactory, 37 unsatisfactory.

In Table 4 (ratings by chemical group) the metal pipe was rated 32 good, 15 fair and 5 unsatisfactory; the plastic pipe rated 36 good, 8 fair and 8 unsatisfactory.

In Tables 4 and 5, five of the nine non-metallics tabulated have a total chemical resistance greater than Type 316 stainless steel. A sixth, vinylidene chloride, is stated to have an equal chemical resistance index.

Cost Vs Chemical Resistance

Because cost of materials is an essential factor in pipe line specifications, a comparative price study is included. Basis of comparison is standard wall 1½ inch iron pipe size, figured in flanged 10-foot sections. The piping forms investigated were high silicon irons, impervious graphite, polystyrene copolymer, polyvinyl chloride, hard rubber, vinylidene chloride, Buna-N hard rubber, polyethylene, rubber lined, polytetra Fluoroethylene lined, modified phenolic, Hastelloy C, vinylidene chloride lined, glass, glass lined, stoneware, copper, aluminum, mild steel, cast iron, lead butyrate, Monel and stainless steel Types 304, 316, 321, and 347. Table 6 lists the relative costs of these materials in comparison to unit costs for plain mild steel. (This index is based on full consumer price. There will be slight variations in relative standings due to special purchase terms or quantity purchases, etc.)

If cost of materials is taken into consideration, the non-metallics make an even more favorable showing. In the group of materials 1 to 4 times the cost of steel, there are 5 metals plus 10 non-metallics and 2 non-metallic lined pipes. This latter group of 12 non-metallics will handle all of the 289 solutions invetsigated. In the group of materials selling at up to only twice the cost of steel, there are five plastics that had total satisfactory ratings for 226 of the 289 corrosive liquids. The solutions which could not be handled (as far as can be determined from the literature; there were many blank ratings) were the solvents, esters, ketones, strong oxidizing agents and combinations of strong acids and borderline cases at temperatures near the temperature limitations of the materials.

Table 5 gives further information on the cost-corrosion resistance relationship. By adding the cost and total

(Continued on Page 30)

	Acetic Acid (50%)	Alum	Aluminum Chloride	Aqueous Ammonia	Ammonium Sulfate	Aqueous Bromine	C Cl4	Aqueous Chlorine	Citric Acid	Ferric Chloride	Formic Acid	HCl (Dil.)	HCl (Conc.)	HF (Dil.)	Hydrogen Peroxide	HNO3 (Dil.)	Phenol	Phosphoric Acid (Dil.)	Potassium Carbonate	Potassium Chloride	H2 SO4 (Dil.)	H2 SO4 (Conc.)		
Ace-Ite	U	S	S	S	S	U	U	S	S	S	S	U	S	S	S	S	S	U	S	S	S	S	U	
Ace Hard Rubber	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	U	
Ace Farian	U	S	S	S	S	U	U	U	S	S	S	S	S	S	S	S	S	S	S	S	S	S	U	
Ace Riviolor	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	
Ace Saran	S	S	S	U	S	U	S	U	S	S	S	S	S	S	S	S	S	S	S	S	S	S	U	
Ace Tempron	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	U	
Aluminum	S	S	?	S	S	U	S	U	S	U	U	U	U	U	S	U	S	U	U	S	U	S		
Butyrate	U	S	S	U	S	U	U	U	S	S	S	U	U	U	S	U	U	S	S	S	S	U		
Cast Iron	?	U	U	S	S	U	S	S	U	U	U	U	U	U	S	U	S	U	S	U	S	U		
Copper	S	S	S	U	S	?	S	S	S	U	S	U	U	S	S	U	S	S	S	S	U	S		
Glass	S	S	S	S	S	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S		
Hastelloy "C"	S	S	S	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S	S	S		
Haveg	S	S	S	S	U	S	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	U		
High Silicon Irons	S	S	S	S	S	U	S	S	S	U	S	S	U	U	S	S	S	U	S	S	S	S		
Impervious Graphite	S	S	S	S	S	U	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		
Chemical Lead	U	S	S	S	S	U	S	U	U	U	U	U	U	U	U	U	U	S	U	S	S	S		
Monel	S	S	S	S	S	U	S	U	S	U	S	S	U	S	S	U	S	S	S	S	U	S		
Stoneware	S	S	S	U	S	S	S	S	S	S	S	S	U	S	S	S	S	S	S	S	S	S		
Steel (Mild)	U	U	U	S	S	U	S	U	U	U	U	U	U	S	U	S	S	U	S	S	U	S		
Stainless, Type 304	S	?	U	S	S	U	S	U	S	U	S	U	U	U	S	S	S	S	S	S	U	U	S	
Stainless, Type 316	S	S	?	S	S	U	S	U	S	U	S	U	U	U	S	S	S	U	S	S	U	U	S	
Stainless, Type 321	U	?	U	S	S	U	S	U	S	U	S	U	U	U	S	S	S	S	S	H	U	S		
Stainless, Type 347	U	?	U	S	S	U	S	U	S	U	S	U	U	U	S	S	S	S	S	S	U	S		
Teflon	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		

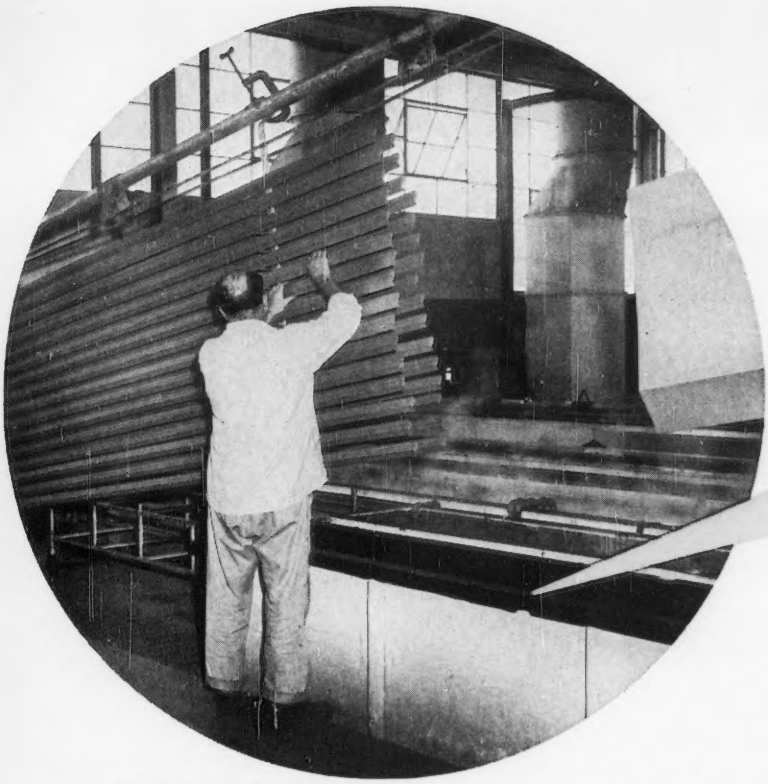
Figure 1—Comparative corrosion resistance of materials in 22 solutions. G = Good, F = Fair and U = Unsatisfactory.

	Salt Solution	Soaps & Detergents	Alkali	Dilute Acid	Sulfates	Bleach Solutions	Animal & Veg. Oils	Caustic Solutions	Gasolines, Fuel Oils	Alcohol & Glycol	Strong Acids	Organic Solvents	Esters & Ketones
Polyethylene	G	G	G	G	G	G	F	G	U	U	G	U	U
Polystyrene Copolymer	G	G	G	G	G	G	G	G	F	F	F	U	U
Polyvinyl Chloride	G	G	G	G	G	G	G	G	G	G	F	U	U
Vinylidene Chloride	G	G	G	G	G	G	F	G	G	G	G	F	F
Carbon Steel	U	G	F	F	F	U	F	F	G	G	U	G	G
Stainless Steel (18-8)	G	G	G	F	G	G	G	G	G	G	F	G	G
Cast Iron	G	G	G	F	G	U	G	G	G	G	F	G	G
Copper	G	F	F	F	G	F	G	F	G	G	U	F	G

Figure 2—Corrosion resistance of 4 plastics and 4 steels to 13 groups of chemicals. G = Good. F = Fair and U = Unsatisfactory.

TABLE 5—Practical Indices for Chemical-Resistant Plastics

MATERIAL	Cost	Sp. Gr.	Struct. Strength	Resist. to Phys. Damage	Resist. to Temp.	Total Prac. Index	Solvents	Salts	Alkalies	Acids	Oxidat.	Total Chem. Resis.	Total Index
Polyethylene	7	9	3	10	1	30	5	10	10	10	7	42	72
Styrene-Acrylonitrile	7	8	6	4	3	28	4	10	6	9	3	32	60
Type II Unplasticized Polyvinyl Chloride	5	8	6	8	3	30	5	10	10	10	7	42	72
Type I Unplasticized Polyvinyl Chloride	5	8	7	4	3	27	6	10	10	10	9	45	72
Vinylidene Chloride	5	7	7	7	4	30	5	10	7	10	6	38	68
Polytetrofluoroethylene	1	6	2	6	9	24	10	10	10	10	10	50	74
Natural Rubber	7	9	4	10	4	34	2	10	10	10	1	33	67
Cellulose Acetate Butyrate	4	8	4	7	3	26	3	7	2	2	1	15	41
Aluminum	7	5	9	6	10	37	10	8	1	2	3	24	61
Steel	9	1	10	6	10	36	10	2	5	1	2	20	56
Stainless Steel 316	1	1	10	6	10	28	10	9	7	4	8	38	66
Pyrex	7	6	5	1	10	29	10	10	2	9	10	41	70



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The extreme corrosive conditions found in metal finishing departments pose some of the toughest coatings problems for corrosion engineers. Here, pickling and plating equipment, as well as structural steel and fixtures, are under constant attack from fumes and spillage of corrosive acids and salts.

Wherever the going is roughest — on tanks, conveyors, ventilators, walls and ceilings — you'll find Tygon is usually the recommended coating. Why? Because Tygon Paint has a proven record of success against corrosion. Tygon lasts longer, continues to give protection in many cases long after other paints have failed, thus costs less in the long run.

Another important factor is that the broad Tygon line permits application of *specialized* protection against a wide range of specific corrosives and exposure conditions. If a vinyl-base coating seems best for the job, one of several Tygon formulations may be specified. If epoxy type coatings are required, Series "E" Tygon Epoxy Coatings offer excellent, long-lasting protection under the conditions for which they are recommended.

Try Tygon on your next stubborn corrosion problem. Bulletin 760 gives the complete Tygon Coatings story. Write for it today.

PLASTICS & SYNTHETICS DIVISION

U. S. STONEWARE

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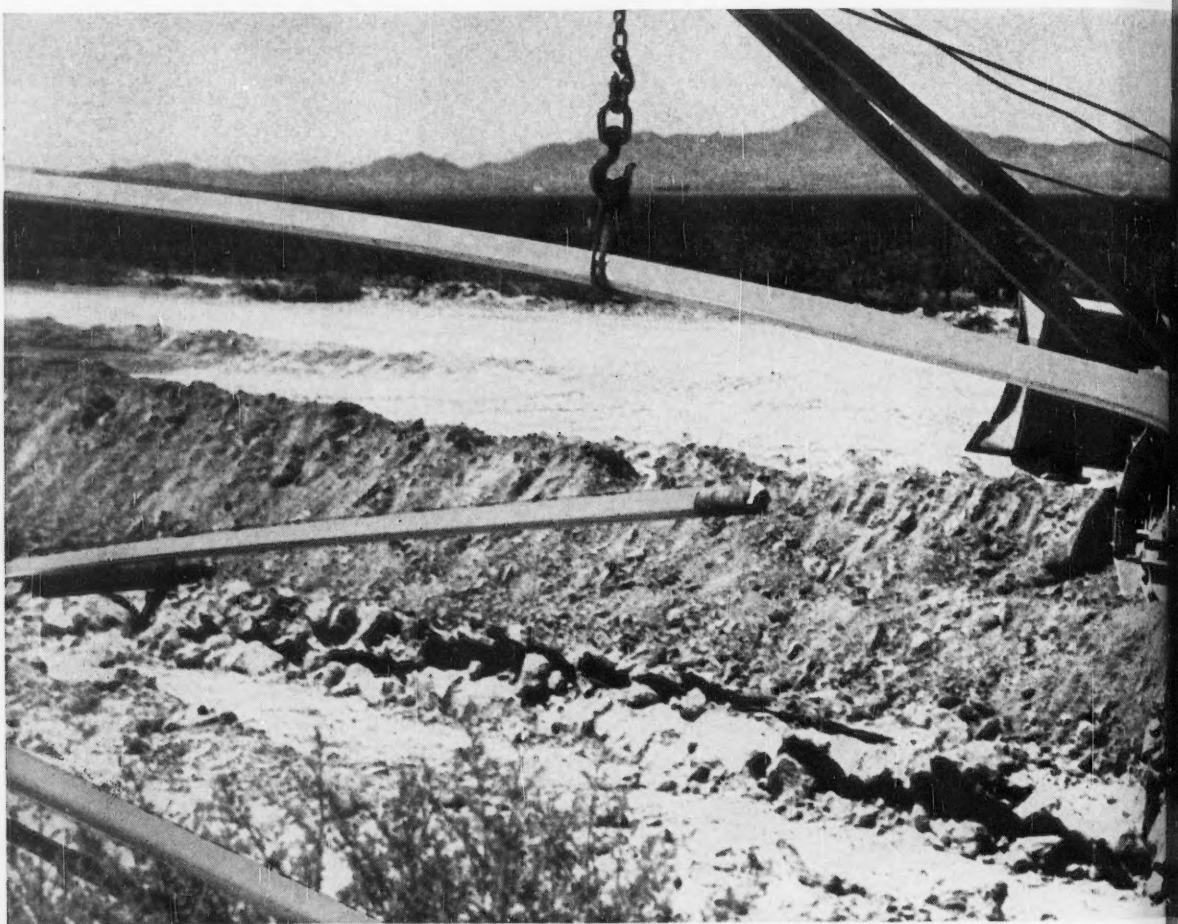
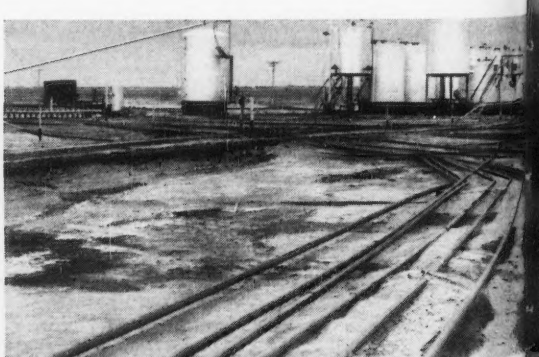
110-G

DECISION:



NO DANGER OF BURN-THROUGH when the line pipe is Republic Electric Resistance Weld. Made from prime, flat-rolled steel. Checked for quality and uniformity as *only* flat-rolled steel can be checked. You get easier bending in field or shop . . . faster line-up of joints . . . better field welds. Mail coupon for details.



EASY WORKABILITY is a characteristic of Republic Continuous Butt Weld Pipe. Uniform strength and ductility assure fast, trouble-free threading . . . excellent, in-service dependability. Tight galvanized coating resists corrosion. Sizes range from ⅛" through 4" nominal. Available in double random lengths. Send for information.

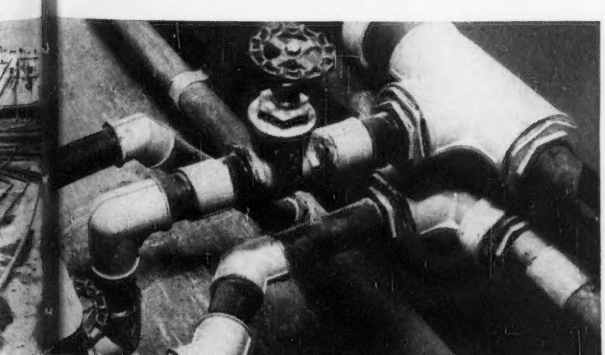
X-TRU-COAT

ARIZONA PUBLIC SERVICE COMPANY IS USING 110 MILES OF THIS PLASTIC-COATED, CORROSION-PROOF PIPE!

With X-TRU-COAT, corrosion will be sealed out *permanently, before* delivery, at *lowest* overall cost. That was the decision of the Arizona Public Service Company, Phoenix, Arizona. They are using *110.7 miles* of this polyethylene-coated pipe in a project designed around company-owned and -serviced irrigation pumps. The project will save farmers as much as 11% yearly on power costs. They will be relieved of excessive investment... pay only for horsepower delivered to the pump shaft.

Anything *but* an ordinary product, Republic X-TRU-COAT merits your investigation. Learn how its tough yet flexible coating wards off electrolytic action... is immune to the most severe acid or alkaline soil... stands up under the abuse of shipping, loading, trucking, and handling. Your Republic distributor has complete information. Call him today or mail the coupon.

See you at the N.A.C.E. Corrosion Show, March 15-17, booth No. 121.



INSTALL WITH CONFIDENCE! The product of a corporation with more than 50 years of pipe-making experience, Republic SRK Plastic Pipe has the high degree of quality essential in severe corrosion applications. Republic has the know-how and facilities — works full-time — to bring you plastic pipe that performs better, longer, at lowest possible cost.

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Non-Metallic Pipe—

(Continued From Page 26)

chemical resistance index to columns, a rating of 39 is established for Type 316 stainless steel. This is exceeded by all the plastics and rubbers except butyrate which is rated 19.

By adding the unsatisfactory ratings of Table 4 to the comparable price index of Table 6, another cost-corrosion resistance comparison can be made. The ideal material would have a value of 1.0 (the lowest cost index of 1.0 plus zero unsatisfactory test results). On this basis vinylidene chloride is rated 1.8; cast iron 2.1; styrene copolymer 3.4, PVC 3.5; copper and steel 4.0; and stainless steel 15.8.

One other possible use of earlier data is the establishment of a cost study based on resistance to all corrosive liquids investigated. These indices, shown in Table 7, consist of the addition of the unsatisfactory rating from Table 3 plus the cost index from Table 6. Again the ideal material would have a value of 1.0 (lowest cost index of 1.0 plus zero unsatisfactory test results). The top 10 positions are held by non-metallics.

Areas of Corrosion where Non-Metallics Excel

Referring again to temperature and pressure limitations, there are still many areas of corrosion that are best handled by the non-metallics. These are the dilute acids, bleach solutions, aqueous halogens, plating solutions, extremely pure water and solutions of ferric salts. In all solutions investigated, however, there were no cases encountered which could be handled only by a non-metallic and not by a metal.

Proper Selection of Non-Metallic Pipe

For a specific service condition, the corrosion engineer usually will have a variety of pipe materials to choose from. In some cases, the service will be such that his choice will be extremely limited.

Wide Range of Choice

A great selection of non-metallic materials are available to handle salt solutions, bleaches, caustics, dilute acids, sulfates, chlorides, plating solutions, photographic solutions, soaps and detergents, animal and vegetable oils, pure water and services requiring non-contaminating pipe. In these cases, cost can be the largest determining factor modified only by temperature limitations and desired physical characteristics.

Regarding these limits, the group of materials selling at approximately 1 to 3 times the cost of steel were compared for heat distortion and working pressures. The maximum short-duration temperature limitations varied from 120 F for polyethylene to 290 F for Buna-N hard rubber. Bursting pressures of the solid non-metallics are quite low in comparison to steel, thus eliminating them from many high pressure services.

For example, consistent quick burst test results for 1½ inch polyvinyl chloride were about 1000 psi, resulting in a maximum working pressure recommendation of about 200 psi.

Further points to consider in the use of non-metallics are availability, prime manufacturer and ultimate vendor. Price should not be too great a factor in selection of a source because there are not many instances of extreme price differentials among similar non-metallic materials. These conditions permit the corrosion engineer to exercise considerable judgment in the selection of manufacturer.

Consideration also should be given to the weight of piping and ease of installation. If there is a large selection of non-metallics suitable for his service, the engineer will find piping that can be flanged, threaded, slip-fit and specially coupled. Of these four types of joints, the solvent weld (or solvent cement) slip-fit connections are usually the easiest and least expensive to install. The disadvantage of this type connection is that the pipe lines cannot be disassembled.

Limited Choice

When solutions such as strong acids, esters and ketones, organic solvents and strong halogens are encountered, the choice of non-metallics is considerably reduced. Temperatures also will be an important limiting factor.

At continuous temperatures of 275 F and below, Buna-N hard rubber can be used for a variety of service conditions and for handling organic solvents, esters and ketones. All the modified phenolics also have heat distortion temperatures greater than the rubbers or plastics. Above these temperature ranges, impervious graphite, polytetrafluorethylene glass and stoneware can be used, depending upon manufacturer's recommendations for specific service.

For chlorine service, the data gathered indicated that styrene copolymer pipe will out perform most other non-metallics costing up to twice as much. Handling of aqueous bromine was shown to be best accomplished with hard rubber, Buna-N hard rubber and glass.

Polyvinylchloride, graphite, glass and stoneware were rated from good to excellent in their resistance to concentrated sulfuric acid. Except graphite, they also were recommended for strong nitric acid.

In these similar instances, the choice of manufacturer becomes quite important. Inconsistencies in the behavior of PVC pipe purchased from different sources have been reported.[4] Apparently, engineering of a non-metallic pipe line should go beyond material specification and include the names of preferred manufacturers.

TABLE 6—Relative Cost of Materials Compared to Mild Steel Unit Costs

Piping Material	Cost Index 1½ Inch (Iron Pipe Size)
Mild Steel	1.00
Cast iron	1.14
Polystyrene copolymer	1.41
Polyvinyl chloride	1.46
Vinylidene chloride	1.81
Polyethylene	1.81
Aluminum	1.92
Butyrate	2.13
Lead	2.35
Glass	2.36
Hard rubber	2.60
Vinylidene chloride lined	2.64
Copper	3.00
Buna-N hard rubber	3.28
Karbate	3.44
Modified phenolic	3.50
Monel	3.64
Rubber lined	3.99
Duriron	5.75
Stoneware	6.14
High silicone iron	9.91
Type 304 stainless steel	9.98
Type 347 stainless steel	12.89
Glass lined	13.06
Type 321 stainless steel	13.14
Polytetrafluorethylene lined	13.76
Type 316 stainless steel	15.83
Hastelloy C	38.43

Summary

Twelve metallic and 12 non-metallic pipe materials were investigated and comparisons made of corrosion resistance and cost versus corrosion resistance. This investigation was a survey of manufacturers' buletins and other publications given as references. It was established that, as a group, the non-metallics exhibit a wider coverage of liquid corrosive services. It was also established, on the basis of full consumer pricing, that non-metallics as a group cost less than the metallics and perform equally well in corrosive services.

The groups of chemicals handled best by non-metallics were supplied as well as references to temperatures and pressures, methods of selecting non-metallics and a short discussion on manufacturer designation.

The overlapping among non-metallics allows the corrosion engineer to select the least expensive, or most practical or most generally available materials for a broad range of corrosion resistance. Literature should be kept up-to-date and checked periodically. Many of the older non-metallics indicated exceptional value; newer forms of plastics had the advantage of lighter weight and ease of installation.

There are very definite temperature and pressure limitations for most non-metallics. Within these limits non-metallic pipe line can be considered for use. For some materials, pressure limits can be extended by use of lined steel pipe.

All solutions investigated could be handled by at least one solid metallic pipe. In many cases, however, cost of the metals would make them impractical except for special service conditions.

References

1. Chemical Engineers Handbook. Third Edition. McGraw-Hill, New York City.
2. Pipelines to the Future, A Study of Plastic Pipe. Monsanto Chemical Company. December, 1955.
3. R. B. Seymour and R. H. Steiner. Plastics for Corrosion-Resistant Applications. Reinhold Publishing Corp., 1955.
4. H. E. Atkinson, Jr., and T. F. Degnan. Erratic Performance of Type 1 Unplasticized Polyvinyl Chloride in 93 Percent Sulfuric Acid Pipelines. *Corrosion*, **14**, 261t (1958) June.

TABLE 7—Relative Standings of Corrosion Resistant Where Cost Is a Factor*

Piping Material	Index of Cost Corrosion Resistance
Polyvinyl chloride	1.5
Glass	3.4
Buna-N hard rubber	4.3
Impervious graphite	4.4
Hard rubber	5.6
Vinylidene chloride	5.8
Modified phenolics	6.5
Polyethylene	6.8
Polystyrene copolymer	7.4
Stoneware	8.1
Copper	9.0
Monel	9.6
High silicone irons	10.8
Aluminum	12.1
Butyrate	12.9
Cast iron	13.1
Polyterafluorethylene	13.8
Lead	14.4
Mild steel	15.0
Type 304 stainless steel	18.9
Type 347 stainless steel	21.9
Type 321 stainless steel	22.1
Type 316 stainless steel	23.8
Hastelloy C	39.4

* Ideal material would have a value of 1.0.

63 Technical Committees to Meet in Dallas

Corrosion problems in various industries will be discussed during the 63 technical committee meetings scheduled to be held during the 1960 NACE Conference, March 14-18, in Dallas, Texas. Problems in oil and gas well equipment, pipelines, utilities, process industries, protective coatings and the refining industry will be included.

All conference registrants are invited to attend any technical committee meeting that will be discussing subjects of interest. Meetings have been scheduled so that committees and technical symposia discussing the same or similar subjects will not meet at the same time. Attempts also have been made to avoid conflicts of technical committees on similar subjects meeting at the same time so that persons interested in a given corrosion problem will be able to attend all committee meetings and symposia dealing with that problem.

Reorganization of Technical Committee T-1 on Corrosion in Oil and Gas Well Equipment will be submitted for approval by the Technical Practices Committee. This reorganization has been proposed to broaden the scope of T-1 unit committees so that people of similar interests will be kept together in committee work, rather than having to attend several unit committee meetings. Proposals have been designed to avoid overlapping of unit activities.

Corrosion of Equipment Discussed at T-8 Meeting

Corrosion in reforming and desulfurization units, exchangers, condensers and high temperature furnace tubes was included in discussions at the meeting of Technical Unit Committee T-8 (Refining Industry Corrosion) held during the South Central Region Conference in Denver, October 13-14.

Other topics discussed were naphthenic acid corrosion, metallurgy phenomenon of interest to corrosion engineers, waterside corrosion problems and corrosometer applications.

Attendance for the meeting totalled 84—representing 55 percent of the committee's active membership.

A meeting is scheduled for Thursday, March 17, during the 1960 NACE Conference in Dallas, Texas.

35 Attend T-1F Meeting Held at Denver Conference

Thirty-five members and guests attended the Denver meeting of Technical Unit Committee T-1F on Metallurgy held during the October 12-15 South Central Region Conference.

Discussion included two reports from task groups, alternate string coupling tests, materials for high temperature production, experimental alloys and stress corrosion cracking in annular fluids.

Chairman R. L. McGlasson appointed H. M. Cooley, R. S. Ladley and L. W. Vollmer as a nominating committee to select a list of officers for the committee.

Painting Technology Requires Proper Personnel Training

Painting has become a highly technical operation that requires proper training for personnel, according to George Stoddard of the De Vilbiss Company, who spoke at the October 14 meeting of Technical Unit Committee T-6D on Industrial Maintenance Painting, held in Denver during the South Central Region 1959 Conference.

Even a simple specification such as "red lead and linseed oil" presents an involved problem, he explained. There are more than 36 different linseed oils varying from pure to 70 percent dilution, and there are at least 16 types of red lead. Type of coatings have become complicated also: alkyds, chlorinated rubbers, epoxy resins, neoprenes, phenolics, polyethylene, styrene copolymers, high solid polyesters of the isolite styrene monomer type, urethanes and vinyls.

Availability of several application methods such as brush, roller and spray further complicate the painting operation.

Painters should be trained so that the advantages of the new coatings can be realized through proper application, he added. A school for painters which is available to companies is operated by the De Vilbiss Company in Toledo, Ohio.

The talk was given as part of the report from Task Group T-6D-6 on Painter Education.

Other task group reports given at the meeting were T-6D-1 on Economics of Maintenance Paintings, T-6D-3 on Painting Programs, T-6D-2 on Painting Specifications, T-6D-4 on Specifications for Shop Cleaning and Priming and T-6D-5 on Painter Safety.

T-2C Chicago Meeting

Evaluation of cathodic protection for bare pipe lines and for concrete coated pipes was discussed at the Chicago meeting of Technical Committee T-2C on Criteria for Cathodic Protection. L. C. Wasson was acting chairman for the meeting; T. J. Lennox, Jr., was recording secretary.

Reports were heard from the two task groups: T-2C-2 (Coupons as a Criteria for Cathodic Protection), and T-2C-4 (Cathodic Protection Criteria Research).

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News deadline for CORROSION is the 10th of each month.

Perry

Boone

Detroit and Chicago Electrolysis Groups Join T-7B Committee

The 22nd and 23rd groups have joined NACE's T-7 Corrosion Coordinating Committee under Unit Committee T-7B, North Central Region Corrosion Coordinating Committee. The two groups are the Detroit Committee on Electrolysis and the Joint Electrolysis Committee of Various Utility Companies of Chicago.

Officers for the Chicago group are Chairman R. I. Perry, Commonwealth Edison Co., and Secretary H. C. Boone, Peoples Gas, Light and Coke Company. They were elected for indefinite terms.

Founded in 1908, the Chicago Joint Electrolysis Committee meets four times each year. About 12 cases are covered at each meeting. A rectifier and bond directory is maintained, and underground records are available with an underground negative system map of the Chicago transit companies. Eighty-five percent of the committee's cases are caused by street car stray current; fifteen percent by rectifier operation.

Gas, water, oil, railroad, telephone, transit, electric and housing companies are represented on the Chicago committee.

General committee officers for the Detroit group are Chairman C. Chapin, Department of Water Supply, Detroit, Vice Chairman P. Hough, Michigan Bell Telephone Co., Detroit, Secretary-Treasurer E. E. Westerhof, Consumers Power Co., Jackson, Mich. Technical committee officers for this group are Chairman R. Forrest, PLC Communications, Detroit, and Secretary D. J. Panetta, Michigan Consolidated Gas Co., Detroit.

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226,800 copies of 25 NACE Technical Committee Reports were distributed in 1957. In 1958, 210,300 copies of 12 NACE Technical Committee Reports were distributed.

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More than 30,000 franchised retail dealers sell motor trucks in the United States.

Committee Officers Recently Elected

T-4H Officers

New officers recently elected by Technical Committee T-4H, Tests and Surveys of Cable Sheaths, are Chairman H. W. Dieck and Vice Chairman R. W. Ackermann.

Chairman Dieck is employed by the Long Island Lighting Co., Mineola, N.Y. He has a BS in electrical engineering from New York University and has been an NACE member since 1950. His work with the utility company is in corrosion control on underground gas pipe, salt water condensers and other equipment.

Vice Chairman Ackermann, an NACE member since 1952, is a corrosion engineer with the Long Lines Department of American Telephone and Telegraph Co., Cincinnati, Ohio. He is a graduate of the University of Minnesota and is a member of AIEE.

T-2 Vice Chairman

R. J. Emerson of El Paso Natural Gas Co., El Paso, Texas, was elected recently as vice chairman of NACE Technical Group Committee T-2 on Pipe Line Corrosion.

Emerson

Before joining El Paso Natural Gas in 1946, he was associated with the metallurgy department of Youngstown Sheet & Tube Company. In his present job, his fields are cathodic protection and pipeline coatings. He has a BS in mining engineering from Texas Western College.

He also is chairman of NACE Technical Unit Committee T-2B on Anodes for Impressed Currents and was chairman of the Pipeline Corrosion Symposium at the NACE National Conference last year in Chicago.

T-4A Chairman

Orville W. Zastrow is the new chairman recently elected by Technical Committee T-4A, Effects of Electrical Grounding on Corrosion. He is associated with the Electric Engineering Division of Department of Agriculture's Rural Electrification Administration in Washington, D. C.

A member of NACE since 1950, he is head of the Equipment Performance Unit in the Operating Problems Section, where his work is concerned with corrosion problems on rural power distribution systems.

T-2K-3 Projects Underway Discussed at Baltimore

Committee T-2K-3 on History and Results held a meeting October 5 during the Northeast Region Conference in Baltimore. F. E. Costanzo, Manufacturers' Light and Heat Co., Pittsburgh, presided. The committee discussed projects under way and results reported so far. The projects are now two years old and must proceed another year before results can be assessed.

The committee also discussed various kinds of plastic coated pipe now available and the best uses for the various kinds with respect to their coatings. These include polyesters, epoxies and polyvinylchloride.

62 Attend Denver Meeting Held an T-1H Committee

Sixty-two members and guests attended the Technical Unit Committee T-1H (Oil String Casing Corrosion) meeting held during the South Central Region Conference in Denver, October 13.

Detection methods, cathodic protection, chemical treatment and three task group reports were included in the committee meeting.

5 Work Units Appointed At T-3G-1 Group Meeting

Five work groups were appointed at the meeting of Task Group T-3G-1 (Cathodic Protection of Hull Bottoms of Ships) held during the Northeast Region Conference at Baltimore, Md., October 8.

The five work groups are (1) Cathodic Protection of Stern Areas of Hulls, (2) Shafts and Propellers, (3) Types of Reference Electrode, Their Positioning and Interpretation of Potential Readings, (4) Materials Available for Use as Anodes and (5) Shielding Required for Both Impressed Current and Galvanic Anode Systems.

Reports from these groups will be given at the T-3G-1 meeting scheduled for March 14 during the 1960 NACE Annual Conference in Dallas, Texas.

Talk on Furane Resins Given at T-6A Meeting

Development and uses of furane resins were discussed by Ed Reineck of Quaker Oats Company, guest speaker at the Denver meeting of Technical Committee T-6A (Organic Coatings and Linings for Resistance to Chemical Corrosion) held in conjunction with the NACE South Central Region Conference, October 13.

Chairman C. G. Munger of Amercoat Corporation reviewed the status of committee reports. Eight reports have been published, four are being revised on rubber linings, phenolic coatings, polyurethanes and Hypalon, four have been received but not sent to the committee for comment and four have not been received.

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NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

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There were 311,758 stockholders in one U.S. steel company in 1958. These stockholders included men and women from all walks of life in every state in the nation.

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In 1958, there were over 11 million privately and publicly owned trucks on U. S. streets and highways, representing 17 percent of the total U. S. motor vehicle registrations.

TECHNICAL REPORTS

on

MARINE COATINGS

T-1M Suggested Coating Specifications for Hot Application of Coal Tar Enamel for Marine Environment. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-8. Per Copy $.50.

T-1M Suggested Painting Specifications for Marine Coatings. A Report of NACE Technical Unit Committee T-1M on Corrosion of Oil & Gas Well Producing Equipment in Offshore Installations. Publication No. 57-7. Per Copy $.50.

Remittance must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 65c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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Membership in NACE Technical Unit Committees

A question frequently asked by guests attending meetings of Technical Unit Committees is "How may I become a member of this committee?" This question is answered in the following procedure quoted from the Technical Committee Operation Manual:

1. Any member of NACE (irrespective of place or country of residence) may become a member of a Technical Unit Committee upon fulfillment of the following requirements.

2. He should apply in writing to the Chairman of the Unit in which he desires membership, stating his interest and willingness to participate in the activities of the Unit and giving his experience in the subject.

3. Approval by the Chairman of the Unit Committee of the application or by majority vote of committee members by letter ballot is necessary for appointment of an applicant to membership in a Unit Committee.

4. The Chairman of the Unit Committee will notify each applicant of his acceptance or rejection.

A directory of NACE Technical Unit Committees and Officers is published in the January, April, July and October issue of CORROSION.

Dallas Conference Program Revisions Made

Kinsolving to Be Corporate Member Luncheon Speaker

William C. Kinsolving, president of Sun Pipe Line Company and Sun-Canadian Pipe Line Company, Philadelphia, Pa., will give the keynote address at the Corporate Member Luncheon to be held Thursday, March 17, during the 1960 NACE Annual Conference in Dallas, Texas.

All NACE corporate members are invited to the luncheon and have been asked to bring a management representative from their companies. Both the member and his company representative will be guests of NACE at the luncheon.

Kinsolving

Mr. Kinsolving joined the Sun Oil Company as an oil field hand in 1923 after receiving a degree in electrical engineering from Cornell University. Advancing through various stages of oil field work, he was placed in charge of construction on the Susquehanna products pipeline from Marcus Hook, Pa., to Cleveland, Ohio, in 1930-31. Later he returned to his native Texas to direct construction and operation of the company's line from the newly discovered East Texas oil field to the Gulf Coast.

In 1942 he was called to Washington to be assistant director of transportation for the Petroleum Administration for War. In that capacity, he was prominent in the planning and construction of the emergency 1300-mile Big Inch Pipe Line from Texas to Philadelphia and New York.

Later commissioned a colonel in the Army Corps of Engineers, he was in charge of laying 1500 miles of pipe line in India, from Tinsukia to Calcutta with a branch line to the long range airfields west of Calcutta. In recognition of this service, Colonel Kinsolving was awarded the Legion of Merit by the U. S. Army and the Order of Yun Hui by the Chinese government.

After completion of his military service in India, Mr. Kinsolving returned to Sun Oil as manager of all pipe line operations. In July, 1947, he was elected president of the Sun Pipe Line Company (Texas) and also of Susquehanna Pipe Line Company. When the two lines were consolidated in 1951, he became president of the new company, Sun Pipe Line Company. His home is in Haverford, Pa.

He was director of the Fifth World Petroleum Congress held in New York City, May 31-June 6, 1959. His area of responsibility included transportation and distribution.

He is a member of the American Petroleum Institute, Texas Mid-Continent Oil and Gas Association, Pipe Liners Club and the Racquet Club, Country Club and Cornell Clug in Philadelphia.

New Abstract Source Covers Russian Topics

A new source of corrosion abstracts has been added to the 21 which provide abstracts for use by the National Association of Corrosion Engineers. This source, The Pergamon Institute, an international organization active in the fields of publication, translation and communication of technical literature, will supply NACE with abstracts in English of articles and books published in USSR, Eastern Europe and China.

Pergamon also will supply NACE with translations of titles of Soviet bloc countries' technical material when the material appears to be corrosion-connected.

Pergamon provides numerous services in the technical translation field: Review volumes of Russian scientific progress. World Technical Press Digest, based on the Express Information Service of the Russian Academy of Sciences. Russian-English scientific and technical dictionaries. A number of Russian journals published in whole or part.

Most of the abstracts received from the institute will be of technical articles available in translation. When this is the case the availability of the translation will appear in the abstract. Pergamon's executive offices are located at 1404 New York Ave., N.W., Washington 5, D.C.

Technical Committee Meetings Changed; Paper Is Withdrawn

Additions and changes in the Dallas Conference Advance Program as printed on Pages 31-57 of the January issue of CORROSION are listed below:

Changes in Technical Committee Meetings

Two committee meetings have been added to those meeting Monday, March 14, at the Adolphus Hotel in Room E. They are T-6C (Protective Coatings for Resistance to Marine Corrosion) and T-6J (Los Angeles Area, Protective Coating Applications Problems).

Technical Committee T-4F-2 (Materials Selection in the Electric Industry) has been added to the two meetings scheduled for Monday afternoon, March 14, Room C in the Adolphus Hotel.

Technical Committee T-6R (Protective Coatings Research) has changed its meeting from Thursday morning to Wednesday afternoon, March 16, in Room 203 of the Dallas Memorial Auditorium.

Technical Committee T-6F (Protective Interior Linings, Application and Methods) has changed its meeting to Thursday morning, March 17, in Room 202 of the Dallas Memorial Auditorium.

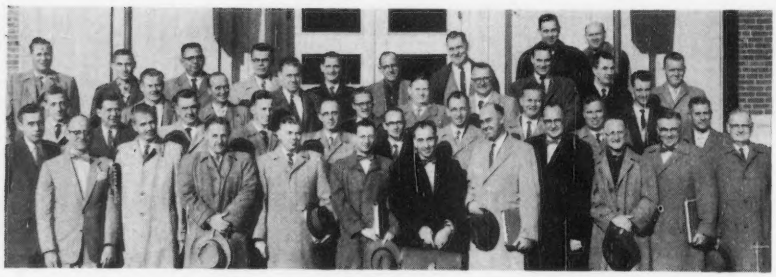
Technical Committee T-6H (Glass Linings and Vitreous Enamels) has changed its meeting place to Room 200 of the Memorial Auditorium. This meeting is scheduled for Thursday morning, March 17.

Technical Program Changes

The paper by Al Erben and R. A. Legault (see Page 38 of the January issue) has been withdrawn. Titled "Cathodic Polarization of Steel in Various Environment," the paper was to be in the Oil and Gas Production Symposium.

A paper entitled "Application of Cathodic Protection to Buried Cables" by D. R. Werner, American Telephone & Telegraph, Kansas City, Mo., has been added to the Utilities Symposium, March 17, 9-11:30 am.

FIVE NACE MEMBERS are among the officers who will plan the 1960 Appalachian Underground Corrosion Short Course. Shown above from left to right, they are T. S. Watson, publications chairman; J. B. Casey, exhibits chairman; C. M. Rutter, Jr., general chairman; J. H. Alm, publicity chairman; R. E. Hanna, secretary and treasurer; and D. A. Tefankjian, program chairman. All are NACE members except Mr. Hanna. The 1960 Appalachian Short Course is scheduled for June 1-3 on the campus of West Virginia University, Morgantown, W. Va.



FACULTY AND REGISTRANTS are shown at the 4th Biennial Short Course on Corrosion Control conducted by the University of Illinois Departments of Electrical Engineering and Mining and Metallurgical Engineering December 7-11 on the campus at Urbana. Total registration was 35 from Illinois, Iowa, Minnesota, Indiana, Nebraska, Missouri, Ohio, Pennsylvania, Michigan and Canada. Banquet speaker was F. W. LaQue, who discussed some of his experiences in the corrosion control field. Three round table discussions were held in the short course.

South Central Region

Tulsa Short Course Plans Completed for Feb. 24-26

Final plans are completed for the 11th Annual Corrosion Short Course for Pipeliners to be held February 24-26 under sponsorship of the Tulsa Section. Two additional instructors have been named recently: Lyle Sheppard of Shell Pipe Line Company and Earl O. Owens of T. D. Williamson, Inc.

Names of other instructors were published on Page 71 of the December issue of CORROSION. A schedule of the course was printed on Page 26 of the January issue.

Registration fee will be $15. Registration forms and additional information can be obtained from Ray Amstutz, Box 4597, Tulsa, Okla.

•

North Texas Section recently elected by letter ballot the following section officers for 1960: Chairman Donald F. Taylor, Jr., of Otis Engineering Corp., Dallas; Vice Chairman Jerry R. McIntyre of Atlantic Refining Co., Dallas; and Secretary-Treasurer George F. Hunt of Johns-Manville Corp., Dallas. These officers were installed at the January 11 meeting. Bob Moberly of Plastic Applicators, Inc., spoke on surface preparation for plastic coating.

•

Tulsa Section officers for 1960 recently elected are Chairman Cecil Smith of Humble Oil Co., Vice Chairman Ray Amstutz of Earlougher Engineering, Secretary, Herb Cooley of Bethlehem Steel Co., Treasurer Jim Rush of Nalco Chemical Corp., and Trustee Dick Lembcke of Cities Service Research.

•

East Texas Section elected its 1960 officers at the December 29 meeting. They are Chairman J. R. Mottley of Tidewater Oil Co., Scroggins, Texas; Vice Chairman Dick Simon of Wesco, Longview; Secretary John Luttig of Atlantic Refining Co., Greggton, Texas, and Treasurer Lee Rutherford of Magna Chemicals, Inc., Kilgore, Texas. A. L. Mortimer of Magna Chemicals, Inc., spoke on the chemistry of amine type corrosion inhibitors.

•

New Orleans Section recently elected its 1960 officers. They are Chairman Joe W. Redden of Napko Corp., Vice Chairman H. R. Hanson of Humble Oil and Refining Co., and Secretary-Treasurer W. B. Ackenhausen of Metairie, La.

Shreveport Section officers recently elected are Chairman R. B. Naremore of Arkansas Fuel Oil Corp., Vice Chairman Grady Howell of Tube-Kote Co., Secretary D. A. Tefankjian of Texas Eastern Transmission Corp., Treasurer Frank Therall of Interstate Oil Pipe Line Co., and Trustee Burt Irish of Irish Engineering Co.

Keith E. James of Halliburton Oil Well Cementing Co., Duncan, Okla., spoke on laboratory research on fluid flowing through a porous media at the section's January 12 meeting. A film was shown on corrosion inhibitor squeeze.

•

Central Oklahoma Section elected its 1960 officers at the December 14 meeting. They are Chairman Frank B. Burns of General Asphalts, Wynnewood, Okla., Vice Chairman Truel E. Adams of Consolidated Gas Utilities, Oklahoma City, and Secretary-Treasurer John A. Knox of Halliburton Oil Well Cementing, Duncan, Okla. Roy Stout of Halliburton Oil Well Cementing Co., Duncan, presented a paper on corrosive characteristics of muriatic acid trapped between tubing and casing.

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West Kansas Section officers for 1960 are Chairman Ray E. Walsh of Atlas Powder Co., Great Bend, Kan., Vice Chairman Bob Adams of Mobile Oil Co., Great Bend, and Secretary Zane Barnett of Tretolite Co., Great Bend.

•

Houston Section had 90 members and guests at the January 12 meeting at which Frank A. Therrell of Interstate Oil Pipe Line Company spoke on the use of deep groundbeds for cathodic protection of pipe lines.

Frank L. LaQue, vice president of International Nickel Co., Inc., New York City, will be guest speaker at the Houston Section's joint dinner meeting February 9 with the Southwest District of the American Society for Testing Materials. This will be the ASTM's annual district meeting and will be held in the Houston Engineering and Scientific Society Building.

Mr. LaQue, president of ASTM and former president of NACE, will discuss "Research in Corrosion."

On February 8, Harris County students selected for awards will be guests of honor at a luncheon to be held at the University of Houston. Mr. LaQue will present these awards and deliver a brief address.

SECTION CALENDAR

February

1 North Texas Section.
2 Shreveport Section. Capt. Shreve Hotel. Cathodic Protection—Design for Interference.
2 Philadelphia Section. Role of the Materials Engineer, by Walter Burton.
4 West Kansas Section.
4 Pittsburgh Section. Modern Architectural Design and Associated Corrosion Problems, by R. A. Fisher, Carnegie Institute of Technology.
9 Houston Section. Joint meeting with Southwest District ASTM. F. L. LaQue, International Nickel Co., Inc., ASTM president, will talk on "Research in Corrosion."
9 San Francisco Bay Area Section.
9 Montreal Section, Cement Corrosion.
16 Chicago Section. Corrosion Roundtable Discussion.
16 Baltimore-Washington Section. Corrosion Problems in Highway Maintenance, by W. J. Halstead.
18 Detroit Section. Joint Meeting with Electrochemical Society. Park Shelton Hotel. Electrochemical Mapping of Plated Surfaces, by H. T. Francis, Armour Research Foundation.
22 Tulsa Section.
23 Panhandle Section.
23 Southwestern Ohio Section. Materials of Construction in Chemical Process Industries, by Harold Boerger, Hilton Davis Chemical Co. To be held in Cincinnati.
25 Edmonton Section.
25 Sabine-Neches Section.

March

1 Shreveport Section. Production equipment. Capt. Shreve Hotel.
3 West Kansas Section.
3 Pittsburgh Section. Design for Corrosion Prevention in the Process Industry, by F. L. Whitney, Jr., Monsanto Chemical Co.
7 North Texas Section.
8 San Francisco Bay Area Section.
8 Montreal Section. Inhibitors.
9 Greater Boston Section. "What We Know and Don't Know About Corrosion," by Frank LaQue, International Nickel.
15 Chicago Section. Lead Sheath Cable.
23 Los Angeles Section.
24 Edmonton Section.
24 Detroit Section. Annual Paint Meeting. R. Gackenbach of American Cyanamid Co., guest speaker.
28 Tulsa Section.
29 Panhandle Section.
29 Southwestern Ohio Section. John Frank, National Distributing Corp., guest speaker. To be in Cincinnati.
31 Sabine-Neches Section.

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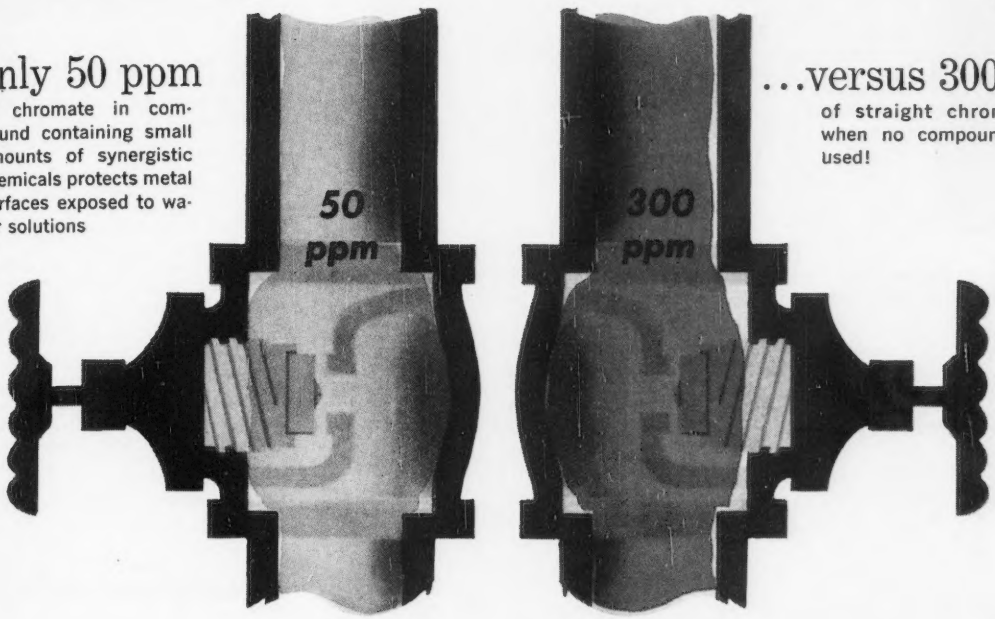

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N. D. Greene, Jr. to Receive 1959 Young Author Award

The 1959 Young Author Award will be presented to N. D. Greene, Jr., at the banquet, March 16, during the 16th Annual NACE Conference in Dallas, Texas.

Mr. Greene is a member of the Department of Metallurgical Engineering, Rensselaer Polytechnic Institute, Troy, N. Y. Formerly he was associated with the Metals Research Laboratories of Electro Metallurgical Co., Union Carbide Corp., Niagara Falls, N. Y. He joined Electromet after receiving his PhD in metallurgical engineering from Ohio State University in 1957. He is a member of NACE, ASM and the Electrochemical Society.

Greene

A total of 23 eligible papers were considered by the Young Author Award Committee.

The award presentation will be made at the banquet by Norman Hackerman, University of Texas, Austin, who is chairman of the committee. Other committee members are Mars G. Fontana, J. J. Harwood, R. B. Hoxeng and Aaron Wachter.

The award, consisting of a gratuity of $50, is made annually to the author or authors of a technical paper published during the year in CORROSION Magazine.

Northeast Region

Niagara Section Plans May Coatings Symposium

A Symposium on Organic Coatings for May 11-13 will be sponsored by the Niagara Frontier Section at Hotel Niagara, Niagara Falls, N. Y. Theme of the symposium will be on the proper use of coatings for protection of ferrous surfaces.

Tentative schedule for the program includes surface preparation talks and demonstrations of various surface preparation methods for May 11. Methods of application with demonstrations will be given on May 12. On Friday morning, May 13, papers will be presented on modern finishes available to fit industrial chemical needs. That afternoon, an informal discussion period will be held at which a panel will discuss industrial painting maintenance problems.

Guest speakers for luncheon and dinner meetings are being planned.

•

Pittsburgh Section scheduled Robert J. Schmitt of U. S. Steel to speak on the role of corrosion testing in the coal-chemical plant for the February 4 meeting.

About 55 members and guests attended the January 7 meeting, at which J. O. Harris of Kansas State University spoke on microbiological studies of pipeline back-filled ditches.

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Philadelphia Section elected officers for 1960 at the annual Christmas Party held December 15. The new officers are Chairman E. D. Bossert, Vice Chairman D. F. Woolley, Jr., and Secretary-Treasurer R. K. Kichline.

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Greater Boston Section heard Lee Sudrabin of Electro Rust-Proofing Corporation speak on applications of corrosion control methods in the Middle East.

Plans for the March 9 meeting include guest speaker Frank LaQue of International Nickel Co., Inc., speaking on "What We Know and Don't Know About Corrosion."

At the May 11 meeting, John Delmonte of Furane Plastics, Los Angeles, Cal., will speak on the possibilities of Furane and other plastics in corrosion control.

•

Southern New England Section saw the film "Mining for Nickel" at the January 6 meeting.

•

Metropolitan New York Section had Thomas P. May, manager of Inco's Kure Beach-Harbor Island Test Stations, as guest speaker at the December 9 Christmas meeting. He spoke on corrosion testing at the stations which he manages.

Southeast Region

Carolinas Section heard Bethel Bond of Rosson-Richards of North Carolina, Inc., speak on a method for increasing voltage in magnesium cathodic protection circuits at the December 4 meeting, held in Charlotte, N. C.

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Miami Section officers for 1960 were elected recently. They are Chairman Newt Bollinger, Peoples Gas Company, Vice Chairman Charles H. A. Montague, Peoples Gas System, and Secretary-Treasurer James J. Abril, Florida Power and Light Company.

•

Birmingham Section has tentatively scheduled four meetings for 1960. The February 4 meeting was planned to have H. W. Ross speak on new types of coatings.

May 6: T. G. Humphreys, Jr., of Alabama Gas Corp., will speak on corrosion above ground.

September 9: J. C. Ray of Southern Bell Telephone will speak on direct distant dialing and will give a demonstration.

November 4: D. D. Holmes of Alabama Power Company will speak on New Power Plants and Associated Corrosion Problems.

1960 officers elected by the section at the December 4 meeting are Chairman John B. Paisley, Jr., Vice Chairman Marion M. Fink, Secretary-Treasurer James C. Ray, and Assistant Secretary-Treasurer Lucien C. Bomar. John A. Waugaman of Good-All Electric Manufacturing Company presented a paper on recent rectifier developments.

FLORIDA GENERAL CONFERENCE gave special honor to Raymond F. Hadley (top right) of Sun Pipe Line Company, Philadelphia, Pa., and Joseph B. Prime, Jr., (bottom right) of Florida Light and Power Company, Miami, at the November, 1959, conference. Mr. Hadley, vice president and chief engineer with Sun Pipe Line, was honored for his contributions and research in corrosion control work. Mr. Prime, an engineer with his company, was honored for his service in helping found the NACE Miami Section and assisting the Florida General Conference. E. J. Tilton, conference general chairman, made the presentations.

North Central Region

Greater St. Louis Section officers for 1960 recently elected are Chairman Walter B. Meyer, Vice Chairman A. Orman Fisher, Treasurer M. C. VanDevanter and Secretary J. W. Cushing. W. A. Heideman and J. Jeter were elected to the section's governing board for two-year terms. Three board members will continue: O. W. Siebert as past chairman and O. H. Fenner and L. O. Goad as board members with one year terms to complete.

•

Kansas City Section elected its 1960 officers at the December 14 meeting. They are Chairman Daniel R. Werner, Vice Chairman H. Shields and Secretary-Treasurer O. E. Moore. Richard M. Fuller presented a paper on current applications of polyvinyl chloride plastics for industrial and utility use.

•

Cleveland Section will hear Mars G. Fontana of Ohio State University speak on unusual corrosion problems at the February 16 meeting.

R. D. Thayer, Jr., spoke on corrosion prevention in masonry construction at the January 19 meeting, held jointly with the Electro-Chemical Society.

Western Region

A. E. Storm of Tidewater Oil Co. Associated, Cal., was elected 1960 chairman of the Western Region. Vice chairman is O. L. Hudrlik of Portland, Oregon; J. W. Caldwell of San Francisco is secretary-treasurer.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1960

February 10-12—Canadian Region Western Division. Vancouver. Hotel Georgia.

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 6-7—10th Annual Western Region Conference, Sheraton-Palace Hotel, San Francisco, Cal.

Oct. 6-8—Southeast Region Conference, Dinkler-Plaza Hotel, Atlanta, Ga.

Oct. 11-14—Northeast Region Conference. Prichard Hotel, Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Schroeder Hotel, Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

Oct. 4-6—Western Region Conference, Hotel Multnomah, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Southeast Region Conference, Miami, Fla., in conjunction with Miami Section's short course.

1962

March 18-22—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 9-11—North Central Region Conference.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

Southeast Region Conference, Birmingham, Ala.

Western Region Conference. San Diego, Cal.

SHORT COURSES

1960

February 24-26—11th Annual Tulsa Section Corrosion Short Course for Pipeliners. Mayo Hotel, Tulsa, Okla.

April 27-29—Portland Section Corrosion Control Short Course.

May 11-13—Niagara Frontier Section Symposium on Organic Coatings. Hotel Niagara, Niagara Falls, N. Y.

June 1-3—5th Annual Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown, W. Va.

September 28-30—Central Oklahoma Section 1960 Corrosion Control Short Course, at University of Oklahoma, Norman.

October 3-5—Corrosion Control Short Course, sponsored by Western Region and University of California.

•

In 1958, there were over 11 million privately and publicly owned trucks on U. S. streets and highways, representing 17 percent of the total U. S. motor vehicle registrations.

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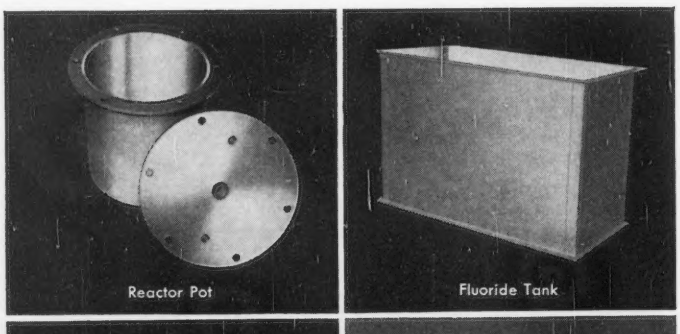

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Hydrochloric, hydrofluoric, and nitric acids, plus the stronger alkalies, oxidants, and solvents cannot harm Teflon Linings. They're easy to clean, non-brittle, non-contaminating, non-flammable, and can be used in **extreme temperatures.**

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Linco, Inc.,
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Mercer Rubber Corp.,
Little Ferry, N. J.

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No Wheels—Less Metals

Is This the Personal Car of the Future?

An all-plastic, no-wheel, single seat personal vehicle may be the cause of a violent upheaval in this country involving highway and bridge construction, traffic control, metals consumption and numerous other areas. Pictured above is a prototype of the Ford Motor Company's version of the wheelless auto—the Levacar Mach I.

Using a cushion of air to raise it from the ground, the vehicle is expected to travel just inches above the nation's streets and highways or above any relatively smooth surface. What effect the general use of these vehicles will have on the nation is a matter of sober speculation in several areas of the nation's economy.

Insofar as highways are concerned, the multi-billion dollar U. S. arterial highway program may possibly be completed just about the time that these vehicles without wheels come into general use.

Ford's Levacar shown above was made of plastic except for the external aluminum shell. The one-piece epoxy lamination for the headrest, back, seat, armrests, floorpan and curved front portion in the dashboard was metallized and chrome plated in the necessary areas. Upholstery for the seat, headrest and armrests was mounted on the plastic.

Because plaster was used on the original clay model to make molds, this Ford prototype body was fabricated from the clay model in less than four weeks. Zinc alloy dies were used for metal forming. Plaster was used for laying up the one-piece epoxy glass cloth lamination for the car's interior. In the more critical areas of the Levacar such as edge moldings with combinations of arcs and curves, epoxy was used in the mold and in the molding formed.

If this prototype model had been built of metal, considerably more time would have been required. One engineer estimated that the plastic model was finished in less time than would have been required just for the tooling to form metal parts needed. Joining the various metal parts and then finishing to hide the joints would have required even more time.

New International Prefixes To Be Used for Multiples

The National Bureau of Standards has decided to follow the recommendations of the International Committee on Weights and Measures to use new prefixes for denoting multiples and sub-multiples of units. The Committee adopted the prefixes at its meeting in Paris in the fall of 1958.

In addition to the eight numerical prefixes in common use, which are given in the table below, the Committee expanded the list by adding the four prefixes marked with an asterisk. Thus, for example, 10^{-12} farad is called 1 picofarad and is abbreviated 1 pf.

Multiples and Sub-Multiples	Prefixes	Symbols	Pronunciation
1 000 000 000 000 = 10^{12}	tera*	T	ter'a
1 000 000 000 = 10^{9}	giga*	G	jig'a
1 000 000 = 10^{6}	mega	M	
1 000 = 10^{3}	kilo	k	
100 = 10^{2}	hecto	h	
10 = 10	deka	dk	
0.1 = 10^{-1}	deci	d	
0.01 = 10^{-2}	centi	c	
0.001 = 10^{-3}	milli	m	
0.000 001 = 10^{-6}	micro	μ	
0.000 000 001 = 10^{-9}	nano*	n	na'no
0.000 000 000 001 = 10^{-12}	pico*	p	pi'co

New Fluorine Compounds

Radiation synthesis for fluorine compounds, a development of Armour Research Foundation of Illinois Institute of Technology, may result in development of new materials. Organic fluorine compounds unobtainable by present techniques may be obtained from controlled radiation reactions. These compounds are valuable because of stability, non-combustibility and good temperature characteristics.

High Strength Steel Is Cadmium Coated By Vacuum Process

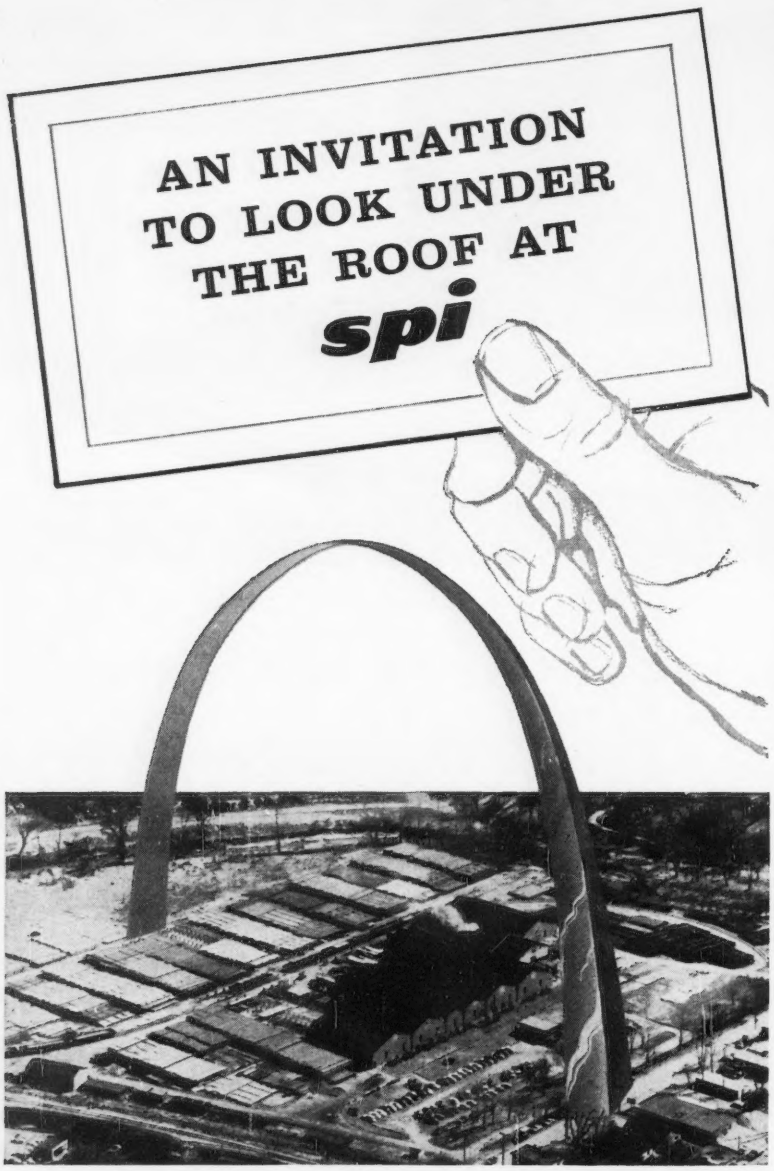
Corrosion protection of highly stressed aircraft components and other parts made of high strength steel is possible through a process for vacuum deposition of cadmium coatings on metal.

This process makes possible the protection of high tensile steel parts against corrosion without subjecting them to the dangers of hydrogen embrittlement. Hydrogen picked up from the acid bath during electroplating has been identified as one cause of service failures.

In the new process, parts are cleaned and then mounted inside a specially equipped vacuum metallizer. After the metallizer has been evacuated, cadmium pellets are heated to their vaporization temperature. These cadmium vapors condense on the work pieces, and the vaporization is continued until the desired thickness has been produced, which usually takes from 15 seconds to several minutes. Thicknesses of more than 0.006 inch have been obtained, but for most applications, best corrosion resistance is obtained if the freshly coated parts are immersed in a chromate conversion solution.

Products being vacuum cadmium coated are aircraft and missile components (particularly high strength bolts, landing gear parts and wing hardware) and high performance structural parts and springs for the automotive, marine and associated industries.

Poly-Kote, Inc., North Attleboro, Mass., and Electro-Vac Division of Radio Cores, Inc., Melrose Park, Ill., have been authorized and equipped for this vacuum cadmium plating service by NRC Equipment Corp., Newton, Mass.



This is an invitation to all oil men, gas men, contractors and utility men to visit our plant in St. Louis at any time. We will be happy to let you look under our roof . . . to conduct you on a tour of our plant to show you our operations.

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Method of Making Nuclear Power Cheaper Explained

Another step to bring cheap nuclear power closer to reality was explained in a paper given at the annual meeting of ASME in December.

The paper explained a proposal to eliminate the huge metal domes that now surround nuclear power plants by substituting a pool of cold water and a much smaller dome. This would cut costs and help to make power more economical as well as reduce any possible hazard from escaping fission products.

In current practice, steel domes are used to protect the atmosphere and the neighboring community in the unlikely event of an accident in the power reactor. The domes have to be large and strong enough to hold all escaping energy from any possible accident, including a ruptured steam line.

In the system described in the technical paper, the reactor would be placed in a pool of cold water. In case of a leak, steam would condense as it hit the cold water, thereby eliminating the need for the big dome. Escaping nuclear fuel or other radioactive material would be contained in the water pool, reducing the chance that any might escape.

NBS Corrosion Progress Reports Published in 1959

Three papers on progress of the work being conducted under the auspices of the Corrosion Research Council at the National Bureau of Standards have been published during 1959. They are listed below.

1. Oxide-Films formed on Copper Single Crystal Surfaces in Pure Water—Part 1. Nature of Films Formed at Room Temperature. By Jerome Kruger. *J. Electrochem Soc*, **106**, No. 10, 847-853 (1959) Oct.
2. Fundamental Corrosion Reactions at the Surfaces of Metals in Selected Environments. By Jerome Kruger. NBS Report 6582. October, 1959.
3. Mechanism of Stress Corrosion Cracking of Metals. By Hugh L. Logan. NBS Report 6581. October, 1959.

A short review of Report 6581 was published in CORRISION, November, 1959, Page 89.

New Polyolefin Plant

A new plant for production of oriented polyolefins at Port Neches, Texas, will be built by Goodrich-Gulf Chemicals, Inc. Initial production will be of high density polyethylene not now manufactured in the United States. When completed late this year, the plant will have a capacity of 13 million pounds annually.

To be manufactured by a new process based on Ziegler chemistry, the Goodrich-Gulf polyethylene is designed to have outstanding environment stress crack and thermal resistance suitable for use in bottle, pipe, wire and cable applications.

Varnish Name Changed

Pacific Paint and Varnish Company has changed its name to DeSoto Chemical Coatings, Inc., Pacific Division.

with CARBO ZINC 11*

Carboline's new zinc filled *inorganic* protective coating

- **All-weather application: —20° F. to 150° F., up to 100% humidity.**
- **All-weather service: —80° F. to 750° F.**
- **Excellent resistance to water, brine, high humidity and organic solvents.**
- **Water-insoluble 20 minutes after application.**
- **Cures from inside out . . . requires no curing solution.**
- **Resists undercutting and subfilm corrosion.**

Carbo Zinc 11 can be used without topcoat or as primer with vinyl, Hypalon or inorganic renewable topcoat for color. Apply with brush or spray. In non-immersion service, it can be applied over a commercial blast surface. It's economical, too . . . low material, application and maintenance costs.

Write today for complete information, technical data, uses and samples of this outstanding, easily applied coating . . . Carbo Zinc 11.

*Patent applied for

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Sales engineers in principal cities. Consult your telephone directory.

COATINGS WITH EXPERIENCE

BOOK NEWS

Handbook of Chemistry and Physics. Charles D. Hodgman, Editor in chief. 3472 pages, 4¾ x 7 inches, flexible cloth. 41st Edition. 1959. The Chemical Rubber Co., 2310 Superior Ave., Cleveland 14, Ohio. Per copy, in U.S., $12; elsewhere, $12.50.

A compendium of useful information on chemistry, physics and mathematics brought up to date in this 41st edition. One hundred pages of additional information added since the preceding annual edition.

This volume succinctly gives much of the basic data and information needed by those concerned with scientific and technical problems related to materials and processes. A 43 page index gives ready access to the information.

This volume is so comprehensive that even a listing of its major subdivisions would be tedious. For the practical worker or student, editor, researcher or other person engaged in almost any kind of modern industrial activity the volume should prove extremely useful.

Translators and Translations. Francis E. Kaiser, editor. 60 pages, 8½ x 11, paper. 1959. Special Libraries Association, 31 East Tenth St., New York 3, N. Y. Per copy, $2.50.

Contains a directory of translators, a section on pools of translations, including a summary of their contents; a list of bibliographies of translations and an alphabetical subject index by geographical, language and subject criteria.

Fuel Elements Conference. 303 pages, 8 x 10½, paper. August, 1959. United States Atomic Energy Commission, Technical Information Service. Published by Office of Technical Services, Dept. of Commerce, Washington 25, D.C. Per copy, $3.00.

Twenty-eight of the 44 papers presented at the May 14-16, 1958 Fuel Elements Conference at Gatlinburg, Tenn. are reproduced. Of those not published 15 appear in classified documents and presumably are available to those with appropriate clearances. The conference was held for the purpose of exchanging classified information between contractors and stimulate overall development of fuel element technology at AEC sites.

Data are given on research reactor, production reactor and power reactor technology and on mobile reactors. A session on radiation damage and a panel discussion on future trends in fuel materials is reported also.

The Corrosion Behaviour of the Major Architectural and Structural Metals in Canadian Atmospheres. Summary of Two-Year Results. 96 pages, 8¼ x 10⅞ in. 24 Feb. 1959. Report by Subcommittee C, Atmospheric Corrosion Testing, E. V. Gibbons, chairman. National Research Council, Associate Committee on Corrosion Research and Prevention, Ottawa, Canada. Availability not indicated.

Results of examinations of exposed panels after two years are given. Covered are experimental procedures, results. Metals exposed included aluminum, riveted galvanic couples, ASTM standard steel and standard zinc, low alloy residual, copper bearing and copper-nickel bearing steels; Types 302, 316 and 430 stainless steels; magnesium alloys and rolled zinc. Eight sites are used.

Appendices give detailed weight loss results, weather records, panel identification schedule and membership list of the committee responsible.

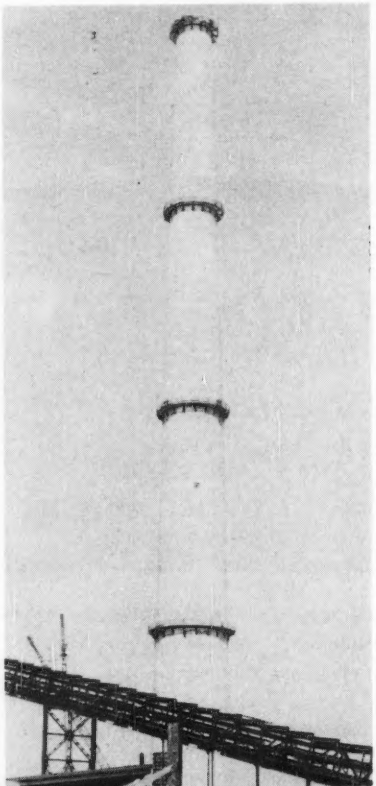
Of materials tested stainless steel is reported to have best corrosion resistance, with aluminum next.

Soldering Manual. 170 pages, 6x9, cloth. 1959. Prepared by American Welding Society Committee on Brazing and Soldering, F. W. Hussey, chairman. American Welding Society, 33 West 39th St., New York 18, N.Y. Availability not indicated.

Principles of sound soldering practice are presented. A table gives relative solderability of metals, alloys and coatings, including flux requirements.

Chapters cover principles, solders, fluxes, joint design, precleaning and surface preparation, equipment, processes and procedures; flux residue treatment, inspection and testing;

Copper and copper alloys, steel, coated steels, stainless steels, nickel and high-nickel alloys, lead and lead alloys, aluminum and aluminum alloys, tin, cast irons, precious metal coatings, primed circuits and safety. There are numerous illustrations and tables. There is a 16 page alphabetical subject index.



NEW FOAMED SILICA MATERIAL was used to insulate this 500-foot chimney from deterioration caused by hot stack gases at a steam generating unit on Staten Island. The chimney was coated internally with acid resistant bituminous compound and two-inch thick blocks of Foamsil, a product of the Pittsburgh Corning Corporation. A half-inch air space was left, and an interior lining of one course of acid resistant brick was erected to protect the Foamsil against abrasion. The silica material is 99 percent pure fused silica glass, according to the producer, and is resistant to all commonly used industrial acids.

Surface Treatment and Finishing of Aluminium and Its Alloys. Second Edition. By S. Wernick and R. Pinner. 607 pages, 5¼ x 8⅜ inches, cloth. 1959. Robert Draper Ltd., 85 Udney Park Road, Teddington, Middlesex, England. Per copy, postpaid $13.25.

The second edition of an encyclopedic work. Data are extensively presented in 204 figures and 120 tables. Each section is accompanied by a list of references.

Chapters cover corrosion and protection of aluminum and its alloys, mechanical surface treatments and finishes, electrolytic and chemical polishing processes, chemical cleaning and anodic etching, chemical conversion coatings, anodizing of aluminum, general notes and theory, decorative and protective anodizing, hard anodizing.

Coloring anodic oxide coatings, sealing anodic outside coatings, physical and chemical properties of anodic coatings, electrodeposition on aluminum (four chapters); organic finishing, vitreous enameling, metal spraying.

Major British and U. S. aluminum alloys are described in an appendix. There is an extensive subject index.

Nature and Properties of Engineering Materials. By Zbigniew D. Jastrzebski. 571 pages, 5¾ x 9 inches, cloth. 20 Nov. 1959. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N.Y. Per copy, $11.

Basic knowledge necessary for intelligent selection of materials, linking chemistry, physics and engineering practice.

The sections of the book (designed for use as a college textbook) and their contents are as follows: Structure of Matter—Atomic structure and periodic table, interatomic and intermolecular forces, states of aggregation, structure of solids.

Colloids and Organic High Polymers—Colloids, formation and structure of high polymers, specific polymeric materials.

Basic Concepts of Phase Transformations.

Mechanical properties—Elasticity, plasticity and flow, properties related to strength, radiation damage.

Metals—Ferrous, non-ferrous.

Ceramic and Related Materials—Clay, refractories, glasses.

Electrical and Magnetic Properties—Electrical and magnetic properties.

Thermal Properties.

Corrosion—Dry, wet, other forms, prevention and control.

Friction, wear and lubrication.

Cementing Materials and Concrete—Joining processes, inorganic cements, concrete.

Protective Materials—Metallic coatings and cladding, chemical conversion coatings, organic coatings and linings, ceramic protective materials.

The chapter on corrosion is thorough and comprehensive. Considering the space devoted to the subject the author has done an excellent job in presenting the subject for use by students.

The book is referenced at the end of each chapter, and has numerous and very carefully executed illustrations. There is an extensive subject index.

Coatings Can Be Checked Internally With Isotopes

Use of isotopes for radiography has made possible the internal inspection of almost any material without affecting the material. All types of coatings can be inspected by radiography without damage to the coating.

In most cases, inspection is accomplished in less time than required by older inspection methods. And it is cheaper, for example, to discover porosities, slag inclusions or other flaws in a raw casting than it is to discover these defects after money and time have been expended on set-up and machining.

Radiography is used to insure proper welding in the manufacture of aircraft, pipelines, bridges, boilers, ships and similar equipment.

X-rays and isotopes are used in radiography. The advantage of isotopes is that no electrical energy is needed; however x-rays have the advantage of variable power to suit the requirements of a given inspection job. Isotopes can be used in small and confined areas because the average isotope is about the size of a pill.

200-Pound Tungsten Ingots Produced by New Process

Large tungsten ingots of weights to 200 pounds are being produced in a new process by indirect sintering in large, high temperature furnaces and forging at high temperatures.

Ingots of pure tungsten and its alloys are being forged 8 inches in diameter and 8 inches in height from tungsten powder in large capacity presses. Tungsten bars previously have been limited to a maximum weight of about 20 pounds.

Applications for tungsten and its alloys are for parts exposed to very high temperatures, to corrosive attack and to radiation. Recent developments in the missile, electronics and chemical industries have increased the demand for tungsten components which have a melting point of 6170 F.

The new process was developed by the Schwarzhopf Development Corp., 595 Madison Ave., New York, N. Y., and the Metallwerk Plansee in Reutte, Tyrol, Austria.

Refractory Symposium

A two-day symposium on refractory materials and alloys will be held May 25-26 at Wayne State University, Detroit, Mich., sponsored by the Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers.

Papers to be presented of probably interest to corrosion engineers are "Oxidation Behavior of Refractory Metals and Alloys," "Chromium and Its Alloys," "Molybdenum and Its Alloys," "Tantalum and Its Alloys," "Tungsten and Its Alloys" and "Rhenium and the Platinum Group Refractory Metals."

Mobil Name Change

General Petroleum Corporation of Los Angeles, Cal., a Socony Mobil company, has changed its name to Mobil Oil Company. The address will be the same: P. O. Box 2122, Terminal Annex, Los Angeles 54.

NEW

ULTRASONIC THICKNESS GAGE

Now you can measure wall thickness accurately with Sperry's new direct reading ultrasonic thickness gage. Battery-operated, it is small and light enough (10 lbs.) to be carried anywhere — to inspect ship hull plates, bulkheads, storage vessels, high pressure pipe.

Using the pulse echo method, this new Sperry thickness gage works even where surfaces are not parallel or where they are corroded and rough. Easy-to-read meter is directly calibrated in inches, and alarm lamps are provided for go-no-go operation or when testing in dark areas.

There is a full line of ultrasonic inspection instruments available from Sperry, for fourteen years leading designers and engineers of ultrasonic testing equipment and SIMAC automated inspection systems.

Write for descriptive literature.

Sperry Products Company

a division of Howe Sound Company

Danbury, Connecticut

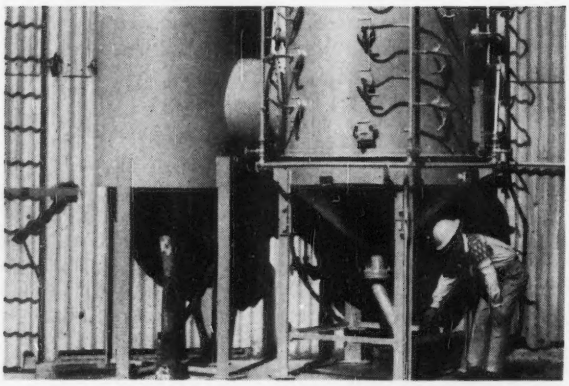
LININGS AND COATINGS OF

NEOPRENE

NEOPRENE is very effective, as either a protective sheet lining or a liquid coating, because of its inherent resistance to a wide range of chemicals, heat, and a variety of other corrosive influences. In processing vessels, neoprene linings handle products that quickly attack linings of natural rubber and many other synthetics: caustic soda, fluorides, ammonium nitrate, greases, oils, fatty acids. Exterior protective coatings benefit from neoprene's excellent resistance to sunlight, weathering and abrasion.

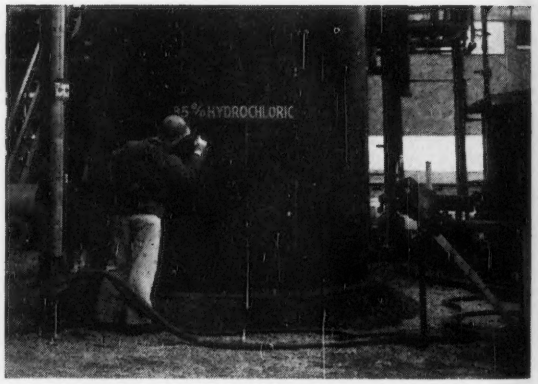
JOB-TESTED TO TAKE T

ABRASION



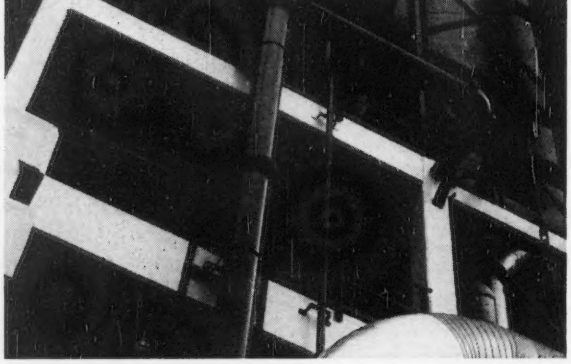
NEOPRENE lining in an acid fume scrubber replaced costly stainless alloys. Abrasive ore dust in acid fumes cut protective oxide film from stainless steel, causing failure in several months. Other organic coatings failed within weeks. Neoprene has been on the job over a year, the best service life on record for this operation.

ACIDS



NEOPRENE maintenance coating was applied in 1950 on an outdoor storage tank for 35% HCl. Recent inspection report by the firm's engineers stated, "To date there have been no repairs to the protective coating. It has stood up remarkably well." Neoprene coating has withstood weather, fumes, and occasional spillage of 35% HCl.

BRACKISH WATER



NEOPRENE pipe linings offered better corrosion resistance than 1/2-inch-thick steel pipe. Brackish cooling water rapidly corroded steel condenser return lines on catalytic cracking units in a petroleum refinery. A 3/16-inch neoprene lining now safely handles corrosive cooling water (18,000 ppm of salt and traces of oil).

CHEMICALS

NEOPRENE tank linings have withstood concentrations of 50-73% caustic soda at temperatures ranging from 145° F. to 250° F. One tank car operator reports an average lining life of 4.9 years — by far the most impressive service on record. Tank car exteriors are now also coated with neoprene.

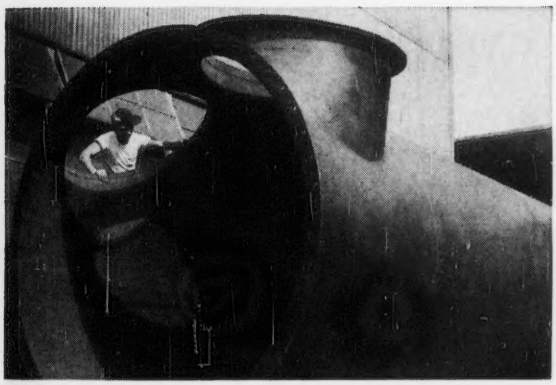
LININGS AND COATINGS OF

HYPALON®

HYPALON meets industry's need for a resilient material with outstanding resistance to strong oxidizing chemicals. Linings and coatings compounded with HYPALON withstand chemicals like hydrogen peroxide, (50%), chromic acid (40%), phosphoric acid (85%), sulfuric acid (93%), nitric acid (20%). These linings perform at temperatures as high as 350° F., offer excellent resistance to abrasion, unmatched resistance to ozone. Exterior coatings are available in attractive colors that resist fading and discoloration.

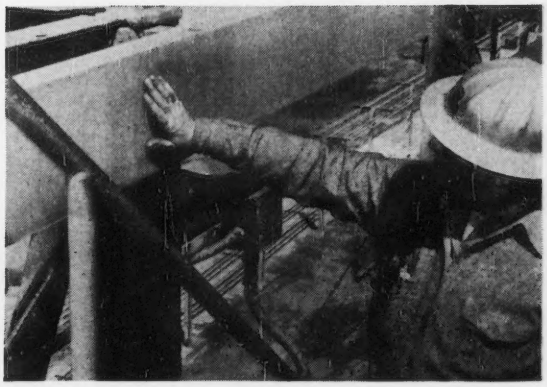
TOUGHEST PUNISHMENT

FLUORIDES



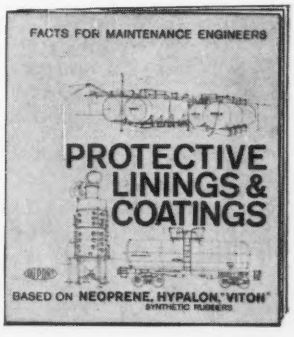
HYPALON Entrainment separator with HYPALON lining receives a mixture of liquid phosphoric acid, gaseous hydrofluosilicic acid and steam from an adjoining vessel at temperatures of 250° F. to 275° F. Lining has been in continuous use for over two years; engineers rate performance "excellent."

CORROSIVE FUMES



HYPALON maintenance coatings in open-air plant protect stairwells and equipment from hot ferric chloride fumes. For 2 years they have kept the structure safe despite fumes, weathering and intermittent temperatures as high as 300° F. Colored coatings permitted engineers to color-code for safety and guard against corrosion at same time.

These are just a few examples of how Du Pont neoprene and HYPALON have overcome corrosion problems. We'll be glad to send you a copy of "Protective Linings and Coatings," a comprehensive brochure designed for the engineer with an equipment corrosion problem. For your copy, write: E. I. du Pont de Nemours & Co. (Inc.), Elastomer Chemicals Department C-2, Wilmington 98, Del.



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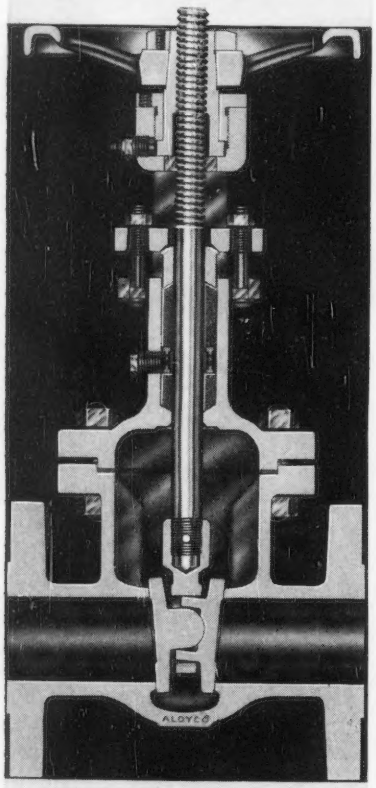
SYNTHETIC RUBBER
NEOPRENE HYPALON® VITON® ADIPRENE®

Better Things for Better Living . . . *through Chemistry*

FOR HIGH-PRESSURE CORROSIVE SERVICE:



300 lb. Aloyco Stainless Steel Gate Valves

Figure 2117 double disc. (above) Available in sizes ½" to 8".
Figure 2217 for solid wedge.
Sizes ½" to 12".
In addition—screwed, and socket weld ends. Sizes ½" to 2".
Other 300 lb. designs including globes, swing and lift checks; jacketed and tank valves.
600 lb. designs also available.

Materials
Types 304 and 304L
Types 316 and 316L
Aloyco 20
Monel and nickel
Hastelloy alloys B and C

For more information on Aloyco valves for your specific corrosive service, write for Bulletin #7 to Alloy Steel Products Company, 1301 West Elizabeth Ave., Linden, New Jersey...the one manufacturer specializing in Stainless Steel Valves exclusively. 9.3

Longer Lasting ALOYCO VALVES
1ST IN CORROSIVE SERVICE

ALLOY STEEL PRODUCTS COMPANY
1301 West Elizabeth Avenue
Linden, New Jersey

Journals Carrying Translations Of Russian Literature Listed

Two journals published in Russia deal exclusively with corrosion. They are:

Akademiia Nauk SSSR. Komissiia po bor'be s korroziei metallov. Trudy. Published in Moscow, beginning in 1951.

Koroziia v khimicheskikh proizvodstvakh i sposoby zashchety ot nee. Published in Moscow beginning in 1954. Classified at the Library of Congress as a monographic series.

According to Charles M. Gottschalk, head of the Reference Section, Science and Technology Division, The Library of Congress, Washington 25, D.C., both are available at the library.

Mr. Gottschalk also said among the many other Russian journals the following frequently publish papers related to corrosion:

Zhurnal prikladnoi khimii. Published by the Academy of Sciences USSR. Library of Congress Call No. TPI.Z63 (Cover-to-cover English translation beginning with v. 23, No. 1, 1950 may be purchased from Consultant's Bureau, 227 W. 17th St., New York 11, N.Y.)

Khimicheskaia promyshlennost'. Published in Moscow by the Ministry of the Chemical Industry, beginning in 1924. Library of Congress Call No. TP1-Z6.

Zavodskaia laboratoriia. Published in Moscow by the Ministry of Metallurgical Industry beginning in 1932. Library of Congress Call No. T4.Z3. (Cover-to-cover translation, beginning with Jan.-Feb. 1958 issue may be purchased from Instrument Society of America, 313 Sixth Ave., Pitsburgh 22, Pa.)

Zhurnal fizicheskeskoi khimii. Published in Moscow, beginning in 1930 by the Academy of Sciences, USSR. Library of Congress Call No. QD1.Z5.

British Sources Given

The following British publications also are a source of translations in the field of corrosion:

LLU Translations Bulletin. Published monthly, York House, Kingsway, London WC 2, England. Annual subscription, 2 pounds sterling, 13 shillings.

Digest of Soviet Technology. Published monthly, Engineering Information Services, Kirkham, Preston, Lancashire, England. Annual subscription, surface mail, $18; air mail $22.

OTS Abstract Sources

Office of Technical Services, U. S. Department of Commerce, Washington 25, D.C. supplies abstracts printed on 5 x 8 inch cards, bound by journal issues of numerous Russian publications. A list of available abstracts can be had on request.

Among the journals for which the abstract cards are available is Kimicheskaya i promyshlennost' and Zhurnal pridladnoi Khimii.

Monthly List of Journals

Miss Lillian A. Hamrick, chief of the Technical Information Division, Office of Technical Services reported that while there is no known list of Russian technical journals dealing exclusively with corrosion, comprehensive coverage of journals in the field is given in the Monthly Index of Russian Accessions, an index prepared by the Library of Congress of the journals received there. The list is available on subscription from Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Subscription price is $12 a year.

Other information in the area of Russian literature sources and translations was published on Page 77 in CORROSION, October, 1959.

Paint and Varnish Clubs Change Name to Societies

The Federation of Paint and Varnish Production Clubs have changed their name to the Federation of Societies for Paint Technology.

The amendment to change the name was passed unanimously at the Federation's council meeting last October.

Purposes of the 37-year old federation are to develop and provide practical and technical facts, data and standards fundamental to the manufacture of paints, varnishes, lacquers, related protective coatings and printing inks and to perform a public service by constant improvement of products and elimination of wasteful methods of manufacture.

Over 3 Million Dollars Spent for NYU Research

Over three million dollars in research expenditures were made for 1959 at the New York University College of Engineering. Financial support was from private industry, government agencies, foundations, professional societies and research funds of the college.

The research program included a wide range of projects in basic and applied research; many of these were concerned with national defense and with problems of public health and sanitation.

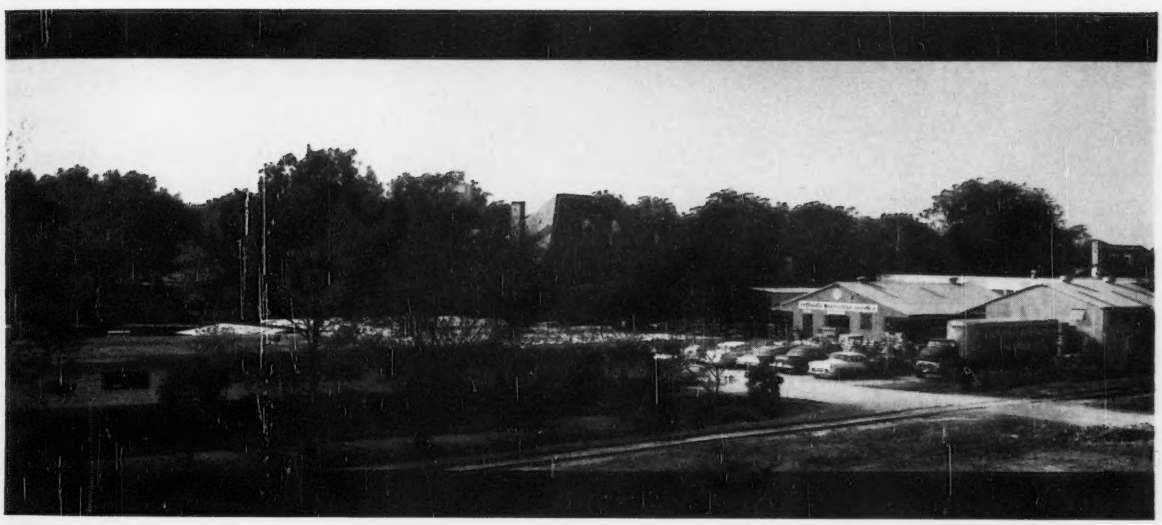
Copies of the 1959 annual report of the NYU Research Division can be obtained from the Office of Information Services, New York University, University Heights 53, N.Y.

Interpolating Thermometer

A platinum resistance thermometer for interpolating between fixed points on the International Temperature Scale (ITS) above 630.5 C, the melting point of antimony, has been developed by the National Bureau of Standards. In addition to simplifying and smoothing the scale, the new thermometer is expected to give greater precision and reproducibility at temperatures to 1063 C (the gold point) than is possible with present thermometric instruments.

CATHODIC PROTECTION SERVICE

YOUR central source for all phases of corrosion prevention



CPS is in a unique position to serve you as a central source for all phases of corrosion mitigation. On any job — new or old — small or large.

CPS offers cathodic protection on a *single packaged* basis. By this we mean, first: CPS furnishes its own engineering crews. They obtain the necessary information to complete the detailed design of a cathodic protection system. Second: CPS furnishes all materials required to complete a job such as: Good-All Rectifiers, CPS-Great Lakes Graphite Anodes, Dow-Magnesium Anodes, Rome Cable and Polyken polyethylene tape to name just a few.

Cathodic Protection Service, this year, is celebrating their 10th Anniversary at the 4601-6 Stanford Street address. Their facilities are pictured above. CPS will be located on the new super Southwest Freeway now under construction. Completion of this freeway will allow CPS to offer faster delivery service to all customers.

Phone, wire or write us concerning how our *single packaged* cathodic protection can provide you cost-cutting advantages on your next job. Finding out will save you money!

Cable Address — CATPROSERV

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TULSA
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RIverside 2-7393

CORPUS CHRISTI
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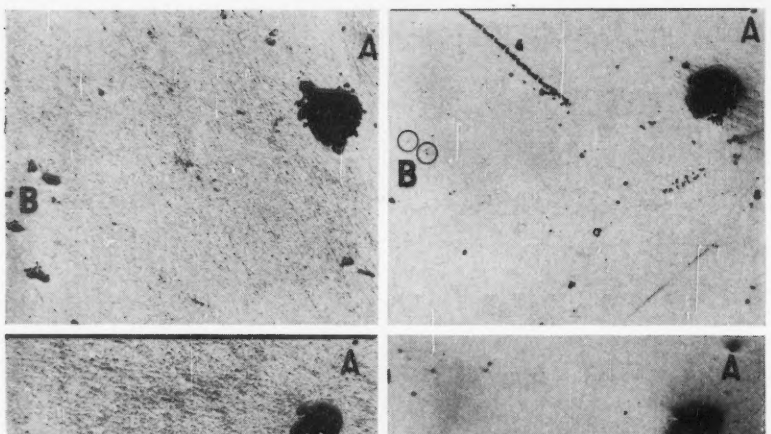
TUlip 3-7264

ODESSA
5425 Andrews Hwy.
EMerson 6-6731



PLATED COATING POROSITY is being studied by microscope by the National Bureau of Standards. In the parallel sectioning technique, three different types of pores found in electrodeposition of coatings are shown above: the continuous pore (A) is larger near the basis metal than at the surface; the dead-end pore (B) extends about one-third of the way through the deposit; and the bridged-over pore (C) goes from the basis metal into the deposit. The four micrographs shown at 400X are successively polished layers from upper left, upper right, lower left, to lower right.

Parallel Sectioning Method Used to Study Coating Pores

Parallel sectioning, a microscopic technique for examination of pores in metallic coatings, has been developed by the National Bureau of Standards. The protective value of a plated coating on metal is dependent, to a large extent, on the exclusion of pores and other flaws in the coating.

Considerable research has been directed toward pore detection and elimination, but methods of examining pores on metallic coatings with microscopes have received little attention because most pores have diameters too small for adequate sectioning. Even if a pore is sectioned properly, the contours may be distorted by polishing and etching. So far, attempts to cut an entire pore parallel to its axis have been unsuccessful; usually only a dot or oval is all that is seen.

In the new NBS parallel sectioning technique, some of the coating perpendicular to the axis of the pore and parallel to the basis metal is polished off. When the cut section is studied under a microscope, the pore appears as a small dot which can be easily seen at 400X magnification if the pore is larger than 0.05 mil in diameter.

To make sure that the dot is not a surface defect introduced during polishing, successive layers are removed and examined until the underlying metal is reached. If the dot is a pore, it will reappear consistently at the same spot. A pantograph arrangement is used to relocate the exact position of the pore after successive removal of each layer.

With this technique, size and shape of pores or other defects that are difficult to cross section can be determined and the damaging effects of atmospheric corrosion can be studied further.

Four types of pore defects have been found in these NBS studies: pits (dead-end pores), cavities (enclosed pores), continuous pores and bridged-over pores. Relative frequency of these four types has not been determined.

Formulator Has Greater Control of Epoxy Coatings

A new method for production of epoxy ester coatings will give the coatings manufacturer control of the molecular weight and length of the polymer chain. Previously, production of this class of epoxy coatings was limited to the use of esterification grade solid epoxy resins. The manufacturer had control only of the choice of acid and degree of esterification.

Using only a liquid epoxy, ERL-2774, bisphenol A and any drying acid, the formular now can prepare in two simple steps a varnish with pre-selected properties.

Coatings formulators and varnish producers will be able to prepare tailor-made epoxy resin products for special applications, according to Union Carbide Plastics Company, Division of Union Carbide Corp., New York, N. Y., developers of the new formulation method.

•

7464 copies of NACE Technical Committee Reports published in CORROSION were sold in 1958.

Drainline System of Glass Used in Chemical Disposal

A new lightweight glass drainline system featuring simple, one-piece couplings for making quick, permanent compression joints is being used for disposal of chemical wastes. The drainline has been designed to cut installation time and cost and to eliminate failure because of corrosion or joint leakage. Engineered for vertical or horizontal mounting, it can be buried in the ground or set in concrete.

Piping and fittings in the system are made of hard, low expansion borosilicate glass that is resistant to virtually all corrosives, but massive quantities of hydrofluoric acid and hot alkalies will affect the material. The coupling consists of a stainless steel shell, a rubber sleeve and a liner of polytetrafluoroethylene.

The drainline system is designed to handle working pressures to 15 psig and temperatures to 250 F, according to the manufacturer, Corning Glass Works, Corning, N. Y.

Brussels University Gives 3 Courses on Corrosion

Three courses related to corrosion control are being given with the cooperation of Centre Belge d'Etude de la Corrosion at the University of Brussels. The courses are as follows:

Electrochemical Corrosion. A 24-hour course with 40 hours of practical work will be given by M. Pourbaix. The course starts 23 January.

Electrochemical Corrosion. A seminar directed by M. Pourbaix at University of Brussels.

Corrosion and Protection of Materials. Including experimental demonstrations, organized in cooperation with the Belgian branch of the Society of Chemical Industry and Belgian Industrial Association. The course was given 18-21 January.

April Building Conference

Surface preparation and field application of paints and coatings will be one of the topics to be discussed at the April 5-7 Spring Conference of the Building Research Institute to be held at the Statler-Hilton Hotel, New York City.

Other topics will include use of adhesives, insulated masonry cavity walls and plastics information workshop.

Heat Extraction Study

A new system of heat extraction from atomic power reactors is being studied at the Center of Information Studies and Experiments in Milano, Italy. The work is based on a contract with the European Community for Nuclear Energy (EURATOM) of Brussels, Belgium.

Anti-Corrosive on Aluminum

A process for eliminating superficial corrosion of aluminum to protect it during shipping, storing and handling has been developed by Harvey Aluminum. The process is said to facilitate anodizing and etching in fabrication.

'60 Copper-Brass Award Deadline Set for March 31

The second annual competition to honor the year's most outstanding contribution to the use, application or metallurgy of copper and copper-base alloys is being sponsored by the Copper and Brass Research Association. The 1960 winner will receive $1000 and an award to be presented May 17 at the Association's annual meeting in Hot Springs, Va.

Entries and nominations for the award must be submitted by March 31. Primary consideration will be given to originality of design of new or established applications of copper, brass, bronze or other copper-base alloys. Entry forms and complete information can be obtained from the association at 420 Lexington Av., New York 17, N.Y.

Wrought Iron Pipe System Melts Snow on Sidewalks

Warming pipes laid in concrete drives and walks eliminate the need of using corrosive deicing salt, shoveling or spreading cinders to provide safe footing during snowfalls. One such piping system recently installed at the Penn-Sheraton hotel, Pittsburgh, Pa., uses ¾-inch wrought iron pipe laid on 14-inch centers. Bleed lines or air vents are welded into the high points of the system to remove air from the lines. Entrapped air, among other things, increases liklihood of corrosion. Other anti-corrosion measures are taken to ensure trouble-free operation of such warming systems.

U. S. Colleges Undertake 10,000 Research Projects

Over 112 million dollars were expended on some 10,000 research projects undertaken during 1959 by 118 leading engineering colleges across the nation. These figures and titles of all projects plus a complete index of research projects are given in the 1959 survey of engineering college research, published by the Engineering College Research Council of the American Society for Engineering Education at the University of Illinois, Urbana, Ill. Copies are $2 each.

Heat Transfer Symposium To Be March 7 in Florida

The University of Florida's Mechanical Engineering Department will hold its annual Heat Transfer Symposium on March 7-8 at the Engineering and Industries Building. Several sessions are planned to include basic principles, temperature and its measurement, heat exchangers, graphical methods, the hydraulic analog and other topics.

Additional information can be obtained from John C. Reed, Mechanical Engineering Department Chairman, University of Florida, Gainesville, Fla.

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More than 30,000 franchised retail dealers sell motor trucks in the United States.

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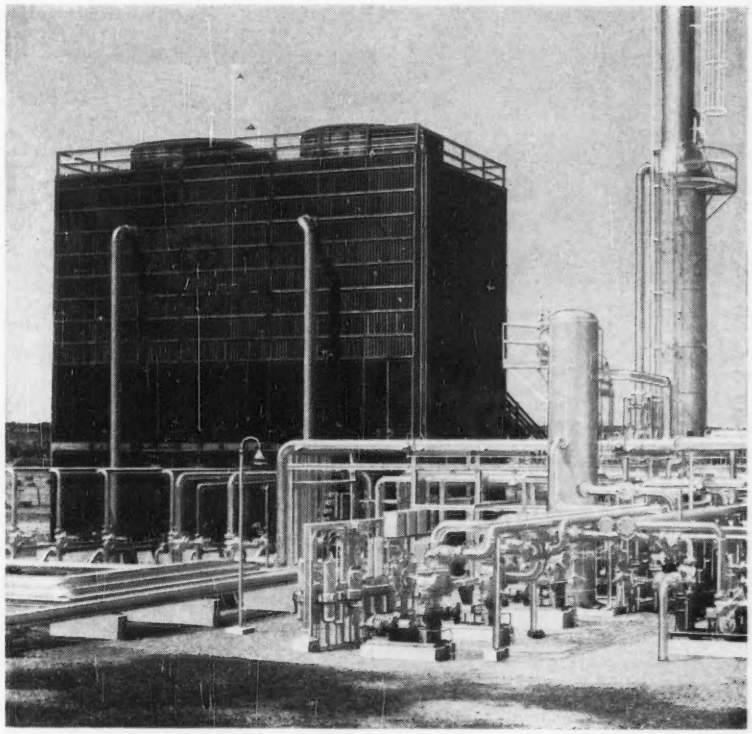
There were 311,758 stockholders in one U. S. steel company in 1958. These stockholders included men and women from all walks of life in every state in the nation.

WANT LOWER CORROSION CONTROL COSTS?



Research and experience with water problems in every kind of industry is why the Calgon Company can save you money by lowering corrosion rates and treatment costs. The service starts with a survey of the system, and includes water analysis, both at the source and in use, a review of flow rates, temperatures, equipment types and usage and other pertinent factors. This is followed by complete treatment recommendations and continuing service after treatment is started. Here is why Calgon methods can save you money and maintenance:

LOWER CHEMICAL COSTS: In most systems, treatment is substantially lower in cost. This is because of more effective treatment, which means that smaller amounts of chemicals can be used.

LOWER WATER COSTS: With more effective treatment, it is frequently possible to maintain higher solid concentrations in cooling water. This means less makeup water, and consequent savings in overall water costs.

BETTER RESULTS: In one typical plant, water treatment in use resulted in a corrosion rate of 4 mils per year. When Calgon methods took over, corrosion rates were reduced to 1 mil per year, troublesome deposits were also eliminated, and chemical costs came down at the same time.

A letter or phone call will put Calgon Engineers to work on your particular problem.

CALGON COMPANY
HAGAN BUILDING, PITTSBURGH 30, PA.

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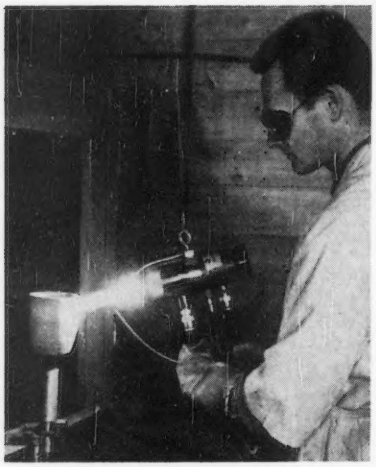


PLASMA FLAME SPRAY GUN develops temperatures of 10,000 to 15,000 F for coating applications of high melting point materials. The gun is shown as a high temperature protective coating is applied to a crucible.

10,000-Degree Flame On New Spray Gun Applies Coatings

A spray gun which develops normal work temperatures of 10,000 to 15,000 F is claimed to spray any material that will melt without decomposing. Using the plasma-arc principle, the gun provides a practical means of applying coatings of high melting point materials such as zirconium carbide, tungsten, tungsten carbide, zirconium boride, zirconium oxide, niobium carbide and other oxides, borides and carbides.

Operating on inexpensive gases such as nitrogen and hydrogen, the gun can generate the necessary heat at about half the cost required with an oxy-acetylene flame. Coating densities can be controlled and approach 98 percent of theoretical. For example, 95 percent dense tungsten and tungsten carbide coatings can be applied to any base material. Physical and metallurgical properties include reduced porosity, improved bond and tensile strengths.

Applications have included spraying of rocket nozzles, missile nose cones and other parts subjected to thermal extremes including crucibles for high melting point materials.

Use of inert gases with the gun eliminates flashback and explosion hazards. Training of operating personnel is relatively simple, according to the manufacturer, Metallizing Engineering Co., Inc., Westbury, Long Island, N. Y.

Laminated Plastic Pipe Is Continuously Produced

Continuous production of laminated thermoset plastic pipe appears possible by a process developed by Armour Research Foundation. Plastic impregnated fabric is fed continuously through a unique arrangement of heated dies to form thermoset plastic pipe of great strength. Fabric reinforcement can be glass fiber, cotton or synthetic fibers; plastic can be epoxy, polyester, phenolic or other thermosetting resin.

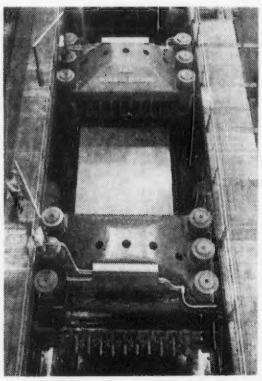
Massive Machine Can Stretch Large Aluminum Sheet 4 Feet

A massive machine capable of stretching a 60-foot piece of thick, high strength aluminum by as much as four feet is being used to refine the internal structure of the metal by equalizing stress distribution in heat treated alloys and welding alloys.

The plate stretcher has a pulling force of 30 million pounds—about twice the capacity of stretchers normally used in the metals industry. It can handle aluminum alloy plate up to six inches thick and 13⅓ feet wide within an over-all cross sectional area limit of 640 aquare inches.

Weldable aluminum alloys stretched on the machine are being used in marine construction, in the chemical process and petroleum industries for tanks and process equipment, in railroad equipment for tank cars and as ballistic armor plate in the lightweight vehicles for the U. S. Army. One application that is growing rapidly is for processing, storage and transportation of liquid methane, oxygen, nitrogen and hydrogen.

Installed in a concrete pit 120 feet long, 32 feet wide and 15 feet deep, the stretching machine is supported on tracks without a guiding frame. No effort is made to hold it in a rigidly fixed alignment. Individual heads are supported on each side by eight railway type car wheels on a double track. Innovations incorporated in the hydraulic system keep the two sides of the machine parallel and completely independent of the load in the gripping jaws.



THIRTY MILLION POUNDS of force can be exerted by this hydraulic machine to stretch 60-foot aluminum plates as much as four feet. It can handle aluminum alloy plate to six inches thick and 13-1/3 feet wide.

The gripping heads are of unconventional design: there is a top and bottom head, each weighing 132 tons, held together by six forged tie bolts, 15 inches in diameter and 15 feet long. Stretching force is provided by 6000 pounds per square inch hydraulic pressure on two 56-inch diameter cylinders.

To absorb the tremendous forces involved should a plate break while being stretched at full power, the stretcher jaw mechanism includes a hydro-pneumatic system similar to the recoil equipment on a heavy cannon.

The stretcher was built by Hydraulik G. M. B. H. of Duisburg, Germany, for Kaiser Aluminum and Chemical Corporation's plant at Ravenswood, W. Va.

New Purifier Produces Ultra-Pure Hydrogen

Low cost production of ultra-purity hydrogen from dissociated ammonia is now possible with a hydrogen palladium diffusion purifier. Impurities are so low that they have not been detected by analysis.

Operating on the principle of hydrogen diffusion through palladium, the unit produces about 70 cubic feet of ultra-pure hydrogen from every 100 cubic feet of ammonia gas fed in. Ninety-three percent of the hydrogen in dissociated ammonia is diffused as purified gas. This pure gas does not contain any measurable traces of nitrogen, inert gases, hydrocarbon, water vapor, sulfur or any of the contaminants usually found in commercial cylinder hydrogen.

The patented purifier was built by the Chemical Division of Engelhard Industries, Inc., 113 Astor St., Newark, N.J.

Literature Program Grant

A grant of $159,200 for a test program to evaluate procedures for exploitation of metallurgical literature has been given to Western Reserve University by the National Science Foundation.

Using a high speed searching selector, the university's documentation specialists will be able to search documents at the rate of 100,000 per hour.

Air Pollution Report

Twenty-nine technical reports are listed in the 1959 Annual Report of the Air Pollution Foundation. These reports have been published by the Foundation.

Various problems of air pollution are covered in the reports listed. The Annual Report is available from the Foundation at 2556 Mission St., San Marino, Cal.

Copper Association Formed

An association of copper producers has been formed to conduct research and studies for expansion of copper and copper product uses and for development of new copper products. Six major copper producers are charter members of the organization which has been titled Copper Products Development Association, Inc.

Additional information can be obtained from Julian Hayes, 25 Broadway, New York 4, N.Y.

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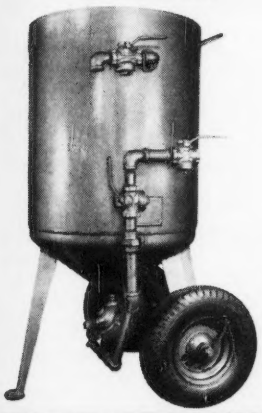
NACE's 16th Annual Conference and 1960 Corrosion Show will be held March 14-18 in the Memorial Auditorium, Dallas, Texas.

CLEMCO SANDBLAST MACHINES



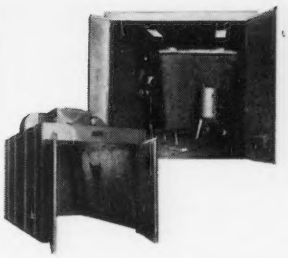
Model SC 1440 — 150 lb. Sand Capacity
Small, stationary, pressure sandblaster. Furnished with automatic pop-up valve, and small sand miser valve. Also available with 6" wheels.

Model SCW 1648 — 300 lb. Sand Capacity
Medium size portable sandblaster. Furnished with automatic pop-up valve, and small sand miser valve. Also available in stationary model.

Continuous Action Sandblasters • Models CA 2456 and CAD 2460
Used by shipyards, refineries, and larger contractors where continuous operation desired. No down time for refilling. Available with one or two sand control valves and with wheels. The highest production on the market.

Model SCWB 2452 — 600 lb. Sand Capacity (Shown)
Most popular size portable sandblaster. Furnished with automatic pop-up valve, and large sand miser valve. Ideal for large contractors or major industries where large volume of blast cleaning is required. (At left).

MODEL SCWB 2452



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Provides 100% safety to your blasters. Instant control at the nozzle will eliminate pot tenders, conserve on compressed air and abrasive. Easy to install on any blast pot regardless of manufacturer.

SUCTION GUN — Model SG 300
Designed for light duty blasting or where limited air supply available. Many advantages: light weight, steady abrasive supply, clean radius and angles, remove rust, paint, mill scale. Uses most common abrasives. No special skill required.

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Equipment Services NEW PRODUCTS Materials Literature

Aluminum

Sheet and Plate Aluminum Alloys with good resistance to corrosive perforation by hot water are commercially available. The new alloys were developed during a 10-year research program conducted by Aluminum Company of America in cooperation with two water heater manufacturers.

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Silicon Aluminum Alloy production will be increased upon completion of the Reynolds Metals Company's silicon plant at its Listerhill Reduction Plant near Sheffield, Ala. The plant's electric furnace operations will have a capacity of about 5000 tons of silicon metal per year, according to Reynolds.

Clad Materials

Insmetals are a group of materials being produced by several companies which have combined facilities and research with International Shielding Industries, Ltd., West St., Wilmington, Del., to undertake consulting, design, engineering, fabrication and construction in shielding against the escape of corrosive, radiation or radioactive corrosives. The metals include Bauxilum (lead surfaced aluminum or alloys), Cupralum (lead surfaced copper or alloy), Ferrolum (lead surfaced carbon steel), Inslead (International Shielding lead), Nicrolum (lead surfaced stainless steel or nickel alloys) and Ferro-Nicrolum (lead surfaced stainless clad steel). A brochure giving technical data on the metals are available from Knapp Mills Inc., 23-15 Borden Av., Long Island City 1, N.Y.

Cleaning

Smooth Surfaces can be obtained by application of a water-like fluid based on colloidal silica to painted surfaces. According to the manufacturer, Du Pont, this soil retardant concentrate fills microscopic pores on the surface and eliminates places for dirt to adhere to. It can be sprayed, brushed, rolled, mopped or wiped on and dries within 15 minutes to a hard, transparent coating.

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Blast Cleaning Structural Steel before use in commercial construction and the economics of this cleaning are discussed in Bulletin 152-D available from Wheelabrator Corp., 1170 South Byrkit St., Mishawaka, Ind. Considerations affecting structural fabricators, construction contractors and owners are discussed with the effects of blast cleaning on maintenance costs and one the life of structural steel coatings.

Coatings, Metallic

Hot Dip Aluminum Coating to prevent atmospheric corrosion and high temperature oxidation and scaling of ferrous parts is described in a brochure available from Arthur Tickle Engineering Works, 21 Delevan St., Brooklyn 31, N.Y. Among applications described are steel corner braces for seaborne aluminum cargo containers to prevent galvanic action and to stop salt spray corrosion, refinery spools, pressure vessel liners and internals, power equipment, heat exchangers and superheater supports. The company's method of metal spray coating of parts too large to hot-dip is also described.

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Galvanized Steel Sheet buyer's manual has been released by the American Zinc Institute, Inc., 60 East 42nd St., New York 17, N.Y. Gives significant factors governing properties, applications, specification, purchase and maintenance of galvanized steel sheet for industrial uses.

Coatings, Organic

Concrete Floor protective coating systems are compared in a new chart released by the Carboline Company, 32 Hanley Ind. Ct., St. Louis 17, Mo. Shows each system's resistance to chemicals, thermal shock, abrasion and temperature. Also gives compressive strength, impact resistance, skid proof properties, thickness and cost per square foot of the individual systems.

•

Aluminum Roof Coating that is claimed to reflect more than 70 percent of the sun's rays is being manufactured by Barrett Division of Allied Chemical Corp., 40 Rector St., New York 6, N.Y. This asphalt-based aluminum coating is designed for renovating old composition roofs and is available either fibrated or unfibrated.

Coatings, Inorganic

Zinkote, a self-curing zinc coating designed to protect bare steel from water, severe weathering, salt spray and abrasion, is described in a bulletin available from Amercoat Corporation, 4809 Firestone Blvd., South Gate, Cal. Applied by regular painting equipment, Zinkote is unaffected by solvents and is not flammable, volatile or toxic, according to the manufacturer.

Filters

Steri-Pore Filters, designed to remove water-borne bacteria, are described in Bulletin 20 available from Filtros Incorporated, East Rochester, N. Y.

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New Lightweight Filters with media elements locked together mechanically are being produced by Purolator Products, Inc., Rahway, N.J. A new high pressure process, using special dovetailed construction, locks filter element parts so securely that the filter medium is embedded in the parent metal of the end caps to form a leak-proof seal, according to Purolator. Because no welding is required, aluminum and dissimilar metals can be used in filter designs.

Triphane Units are being designed to convert existing hydraulic service carts to deliver fluid filtered to 3-microns absolute. The filter-hose-coupling assembly is available in 5000-psi units with capacities from 6 to 30 gpm from Aircraft Porous Media, Inc., a Subsidiary of Pall Corp., Glen Cove, N.Y.

Instruments

Mirror Test for Corrosiveness of printed circuit resin fluxes is described in a bulletin available from London Chemical Co., Inc., Dept. CO-5, 1535 North 31st Ave., Melrose Park, Ill. The bulletin can be used as a reference chart for selecting a proper soldering flux.

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Elecotector, a new electric micro-gauge and comparator, for quick and accurate measurement of coatings thicknesses is being distributed by Ferro Corporation, 4150 East 56 St., Cleveland 5, Ohio. In addition to measuring metallic and nonmetallic coatings on dissimilar bases, the unit also will compare hardness and grades of metals and other materials, according to the distributor.

•

Gas Regulator that is dome-loaded and designed to handle inlet gas temperatures to 450 F is being produced by Western Sky Industries, 21301 Cloud Way, Hayward, Cal. It is adjustable from 200 to 2500 psig and uses a gas-loaded rather than spring-loaded diaphragm to give less pressure variation. Regulator body is made of corrosion resistant steel.

•

Dual Purpose Instrument called the Master is designed as a combination pipe finder-leak detector. Transistorized, it has indepedent circuits for each operation. Additional information can be obtained from Fisher Research Labatory, Inc., Palo Alto, Cal.

Metals, Exotic

Columbia-National Corp., producing zirconium sponge for the Atomic Energy Commission and other users, will be owned solely by Columbia-Southern Chemical Corporation as result of stock exchange with National Research Corp. Formerly National Research owned 50 percent less one share of the Columbia-National. The zirconium plant is in Pensacola, Fla.

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Constitution Diagrams is a bulletin designed to provide convenient, condensed data and selected references on binary and other complex diagrams pertaining to molybdenum. It is available from Climax Molybdenum Company, Division of American Metal Climax, Inc., 500 Fifth Ave., New York 36, N.Y.

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Large Tantalum Sheets, 36 inches wide by 72 inches long, are being produced by Kawecki Chemical Co., Boyertown, Pa. These non-welded tantalum sheets are produced by a newly perfected process.

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Oremet Zirconium Castings are described in specification and data sheets

(Continued on Page 54)

REPRINTS

Of Articles Published In Corrosion

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Add 65c per package to the prices given below for Book Post Registry to all addresses outside the United States, Canada and Mexico. Send orders and remittances to NACE, 1061 M&M Bldg., Houston 2, Texas.

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THREE PAPERS ON SULFIDE CORROSION

A Note on the Value of Ammonia Treatment for Tank and Casing Annulus Corrosion by Hydrogen Sulfide by Walter F. Rogers

Use of Ammonia to Prevent Casing Corrosion by H. E. Greenwell, Rado Loncaric and Harry G. Byars

Electrochemical Studies of the Hydrogen Sulfide Corrosion Mechanism by Scott P. Ewing 1.00

Stainless Steel, Titanium

Effect of Sigma Phase vs Chromium Carbides on the Intergranular Corrosion of Type 316 and 316L Stainless Steel (Part 1—A Survey of the Literature) by Donald Warren ..... .50

The Effect of N_2, HNO_2, and HNO_3 on Corrosion of Stainless Steel by H_2SO_4 by W. P. McKinnell, Jr., L. F. Lockwiid, R. Speiser, F. H. Beck and M. G. Fontana .50

Inhibiting Effect of Hydrofluoric Acid in Fuming Nitric Acid on Corrosion of Austenitic Chromium-Nickel Steels, by Clarence Levae, David Mason and John Rittenhouse ..... .50

NEW PRODUCTS

(Continued From Page 52)

available from Oregon Metallurgical Corp., P. O. Box 484, Albany, Oregon. Major uses for zirconium are in water cooled nuclear reactors where corrosion resistance to hot water is a problem. Zirconium, unlike many metals, does not wastefully absorb the neutrons which trigger the fission reaction.

Non-Metallics

Nocordal Tubes, made of impervious graphite, are being used in a grid type heat exchanger to reclaim zirconium from waste salt cake by a re-dissolving process using hydrochloric acid. Manufactured by Heil Process Equipment Corp., 12901 Elmwood Ave., Cleveland 11, Ohio, the exchangers are used also for dissolving and concentrating solutions of uranium, titanium, beryllium, hafnium, plutonium and other rare metals.

•

Corocrete Terrazzo Flooring material designed for corrosion resistance and high strength is available from Ceilcoate Company, 4832 Ridge Road, Cleveland 9, Ohio. It is recommended for installations where attractive appearance must be combined with chemical resistance to acids, alkalies, oils, solvents, cleaning agents and other chemicals.

•

Impervious Graphite is being used for entrainment separator modules for separation of entrained liquids from corrosive gas streams. Available from National Carbon Co., Division of Union Carbide Corp., 100 East 42nd St., New York 17, N.Y., the corrosion resistant modules are designed for high collection efficiency and low pressure drop, are one by two feet and can be assembled into banks to fit any duct or process vessel.

Pipe

Copper Clad Seamless Tubing and seamless stainless steel tubing are manufactured by Texas Instruments Inc., Metals and Controls Division, 34 Forest St., Attleboro, Mass. Held together by a bond of the same nature as the metallic bond that gives solid metal is strength and ductility, the seamless clad tubing is designed to have high temperature, corrosion resistant and tensile properties.

•

Natco Clay Sewer Pipe is available with Vitri-Seal joints, a compression type seal that is designed to be self-aligning, leak-proof, resilient, acid and alkali resistant and root-proof. The joint consists of polyester fixed rings in the bell and on the spigot ends and a specially designed flexible rubber O-ring which acts as the seal. The pipe is produced by Natco Corporation, 327 Fifth Ave., Pittsburgh 22, Pa.

Plastics

Byers PVC Pipe is described in a new catalog available from A. M. Byers Co., Clark Bldg., Pittsburgh 22, Pa. A special section gives the corrosion resistance ratings of the PVC pipe.

•

Heat-Reactive Vinyl Tubing which contracts at temperatures over 275 F to form skin-tight electrical insulating armor for symmetrical rods, tubes or contoured shapes is produced by Irvington Division of Minnesota Mining and Manufacturing Co., 900 Bush Ave., St. Paul 6, Minn. Called ScotchTite Heat Reactive Tubing, it is designed to shrink under heat to 30 percent in diameter and 15 percent in length in 4 to 8 minutes at 300 F. The tubing is applied over the object to be covered and suspended in any heated chamber.

•

Tube Turns Plastics, Inc., Louisville, Ky., has developed all-Penton and Penton-lined metal fittings and metal pipe with injection-molded Penton lining for handling solvents and hot corrosive fluids in process piping. Penton is the new chlorinated polyether thermoplastic credited with exceptional corrosion resistance and ability to handle elevated temperatures, including low pressure steam. This pipe and fittings can be joined readily by thermal welding of socket-type fittings or by threaded connections with a back weld recommended, according to the manufacturer.

•

Grayguard Mastic Sealant based on Du Pont's Hypalon synthetic rubber is designed for joint sealing and glazing where sunlight, ozone, water, strong oxidizing chemicals, heat, abrasion and movement are factors. Developed by Grayguard, Inc., P. O. Box 1644, Wilmington 99, Del., the mastic bonds tightly to most engineering and structural materials, then self-cures to a resilient rubber seal, according to the company.

•

Estane VC, a new rubbery plastic material made of polyurethanes, has been developed by B. F. Goodrich Chemical Co., Akron, Ohio. Possible uses of the material include wire and cable jacketing, airplane de-icers and coated fabrics for tarpaulins and inflatable products.

•

Delrin Acetal Resin, a new thermoplastic material developed by Du Pont, is available in extruded rods to three inches in diameter from United States Gasket Co., Plastics Division of Garlock Packing Co., 441 Main St., Palmyra, N.Y. The new material is claimed to have outstanding tensile strength, rigidity, fatigue life and resistance to creep and solvents at room and elevated temperatures.

•

High Temperature Applications of chemically inert Fluroflex-T dip pipes, spargers, thermowells and nozzle liners in the chemical and processing industries are described in a new technical bulletin available from Resistoflex Corporation, Roseland, N.J.

Research

Particle Size Analyzer is under development in the radioisotope laboratory of Evans Research and Development Corp., 350 East 43rd St., New York 17, N.Y. Designed to determine particle size and distribution, the analyzer will have applications in the construction, drug, electronics, rubber and other industries which use powders, emulsions, suspensions, gels and aerosols.

•

Research Center Facilities for Minnesota Mining and Manufacturing Co., St. Paul, Minn., will be expanded when a 14-story administration building is completed in 1962.

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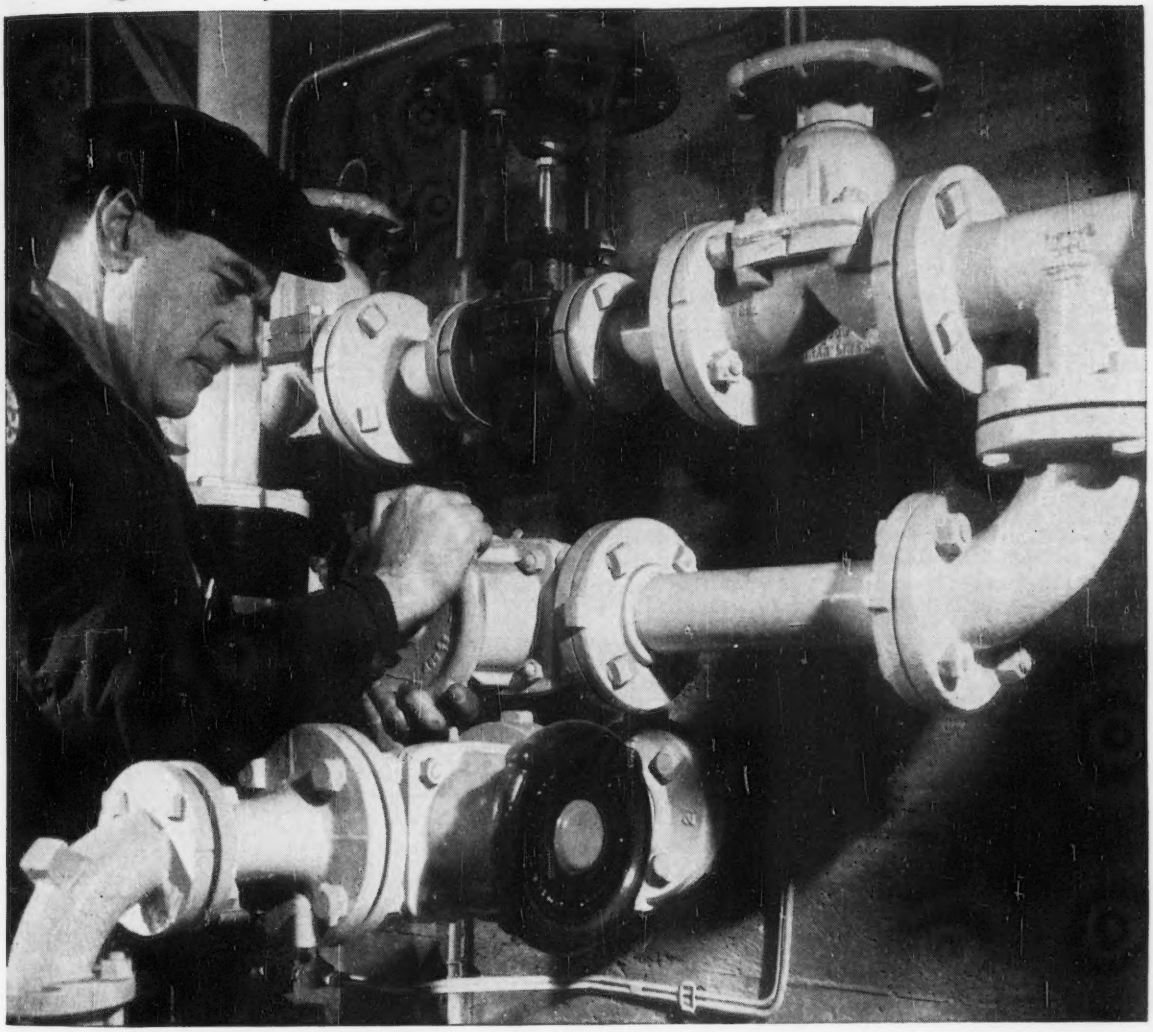

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MEN in the NEWS

N. T. Shideler, an NACE member, now is a coatings advisor. Formerly he was associated with Pittsburgh Coke and Chemical Company.

•

William I. Ballentine, Jr., has been named chief engineer for Southern Pipe Division of U. S. Industries, Inc., Azusa, Cal.

•

Robert A. Barr has been elected a vice president of Babcock & Wilcox Company, to be in charge of the refractories division.

•

George B. Boon has been promoted to technologist with Monsanto Chemical Company's Research and Engineering Division, 800 North Lindberg Blvd., St. Louis 66, Mo.

•

Roy V. Comeaux, NACE member, has been promoted to research specialist in Humble Oil and Refining Company's Research and Development Division at Baytown, Texas.

•

Albert H. Clem has been named vice president of marketing for Pennsalt Chemicals Corp., Three Penn Center, Philadelphia 2, Pa.

•

Everett E. Cox has been appointed Division Superintendent—Titanium for Crucible Steel Company's Midland Works. Replacing him as Superintendent of Production at the Titanium Division is **Jay J. Patten.**

•

T. L. Dahlberg has been appointed manager of maintenance finishes sales by DeSoto Chemical Coatings, Inc., 1350 S. Kostner Ave., Chicago 23, Ill.

•

Earl J. Davis, formerly district manager at Dowell's Denver office, has been named manager of the Houston district, replacing **Jack Talbot,** who has been transferred to the company's industrial operations in Cleveland. **Jack Sutherlin,** former district manager at Wichita Falls, has been transferred to Denver.

•

Shelby A. Davis has been appointed sales manager of the coatings division of U. S. Stoneware Company and its affiliated company, Chamberlain Engineering Corporation.

•

Edward J. Dulis has been appointed to the newly created position of manager of product research at the Crucible Steel Company's Central Research Laboratory.

•

J. R. Callaway has been appointed representative in Dallas, Texas, for the Lunkenheimer Company of Cincinnati, Ohio.

•

G. J. Esselen has been named sales manager of the Industrial Chemicals Division of Pittsburgh Coke & Chemical Company.

•

Arthur J. Freedman, NACE member formerly associated with the Engineering Research Department at Standard Oil of Indiana, has been named project leader of cooling tower research at Nalco Chemical Company, 6216 West 66th Place, Chicago 38, Ill.

•

Wesley E. Gatewood has been appointed director of field sales for the Barrett Division of Allied Chemical Corp., 40 Rector St., New York 6, N. Y.

•

John Geyer has been promoted to technical assistant to the president of Amchem Products Inc., Ambler, Pa. He will be succeeded in the product development department by **Hugh Gehman.**

•

DeWayne Higdon has been appointed South Texas Regional Sales Manager for Tubular Lining Corporation, P. O. Box 20015, Houston 25, Texas.

•

James J. Kelly has been appointed assistant to the vice president of manufacturing of Taylor Fibre Co., Norristown, Pa.

•

Richard S. Latham, partner in the firm of Latham, Tyler and Jensen of Chicago, has been elected president of the American Society of Industrial Designers.

•

C. P. Larrabee, NACE member and former chairman of NACE's Technical Practices Committee, has been appointed chief research engineer in corrosion prevention at United States Steel Corp., 525 William Penn Place, Pittsburgh 30, Pa.

•

Robert F. Mehl, dean of graduate studies and head of the Department of Metallurgy at Carnegie Institute of Technology, has become a consultant for scientific liaison between U. S. Steel Corporation's research organization and various universities and research institute in Europe.

•

John R. Morton has been appointed production engineering manager of the Whittaker Controls Division of Telecomputing Corporation, 915 North Citrus Ave., Los Angeles, Cal.

•

Fredric V. Schossberger has been named assistant director of chemistry and chemical engineering research at Armour Research Foundation of Illinois Institute of Technology, 35 West 33rd St., Chicago 16, Ill.

•

R. E. Seaton has been promoted to manager of processing and power sales at Wolverine Tube Division of Calumet & Hecla, Inc., 17200 Southfield Road, Allen Park, Mich.

POSITIONS WANTED AND AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at $10 a column inch.

Positions Available

Metallurgist, chemist, chemical engineer—ferrous metallurgical background for research and development on high and low temperature corrosion problems of fuel burning equipment. Chattanooga, Tennessee location. Salary commensurate with experience. CORROSION, Box 60-4

Sales Engineer—Progressive company, corrosion and chemical plant experience desirable. Reply CORROSION, Box 60-5.

Maintenance Specialist: Unusual opportunity for engineer to initiate corrosion prevention program. Experience necessary in plant maintenance and paint application. Office suburban laboratory, Minneapolis. Limited travel. Write John Warner, Cargill, Inc., 200 Grain Exchange, Minneapolis, Minnesota.

Sales Engineer—Oldest and largest national manufacturer of complete line of corrosion resistant materials requires man for Louisiana area. Headquarters in New Orleans. Salary and Expenses with car—splendid opportunity—age 28-37 preferred. Include complete personnel and experience resume: CORROSION, Box 60-9.

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Corrosion Engineer. Over 12 years' experience. Will direct cathodic protection department for pipe line, or branch office for progressive firm. Professional engineer. Valuable contacts 5 Southeast states. Also interested as manufacturer's agent-representative. Immediate availability. CORROSION, Box 60-8.

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Large Mid-Western manufacturer of cathodic protection rectifiers is looking for new sales and distribution outlets. Replies strictly confidential. CORROSION, Box 60-10.

CORROSION ABSTRACTS

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2. TESTING

2.3 Laboratory Methods and Tests

2.3.7, 3.5.8

Uni-Directional Axial Tension Fatigue Tests of Beryllium Copper and Several Precipitation Hardening Corrosion-Resistant Steels. M. H. Weisman, J. Mclill and T. Matsuda. Paper before Am. Soc. for Testing Materials, 2nd Pacific Area Nat'l Mtg., Los Angeles, Sept. 16-21, 1956. ASTM Special Technical Pubn. No. 203, Fatigue of Aircraft Structures, 47-60; disc. 61-66 (1957).

Results of notched tensile tests and smooth and V-notched fatigue tests are reported for beryllium-copper, AM350, A-286 and 17-7PH. Cyclic loading tests also are reported for dimpled and riveted sheet specimens of AM350 which simulate typical single-lap riveted aircraft joints. Other tests are reported for 17-7PH steel specimens containing unloaded rivet-filled holes. Purpose of tests was to determine effect of presence of certain types of notches and other stress concentrations on the static tensile strength and fatigue under conditions of uni-directional axial tension loading (stress ratio R equal to zero) over life range of 10^3 to 10^7 cycles of stress. Tables, graphs.—INCO. 15157

2.3.7, 6.2.5, 3.5.9, 3.4.7, 3.5.8

Results of Thermal Cycling Stress Corrosion Tests of Type-347 Stainless Steel in 500 F pH 10-11 Lithium Hydroxide. G. H. Borrmann and G. E. Galonian. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn. KAPL-M-GHB-1, January 8, 1959, 18 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C.

The results of a one-month thermal cycling corrosion test of three stressed T-347 stainless steel bolts exposed to 500 F pH 10 to 11 lithium hydroxide are described. Tests were made to evaluate corrosion of stressed T-347 and T-304 stainless steel under conditions of water leakage to the atmosphere.—NSA. 17775

2.3.7, 6.3.6, 3.5.8, 3.7.2

On the Corrosion-Fatigue Properties of Brasses in a 3% Sodium Chloride Aqueous Solution. K. Takeuchi. J. Japan Metals Inst., **22**, No. 3, 132-136 (1958) Mar.

A series of experiments were carried out for the corrosion-fatigue properties in a 3% sodium chloride aqueous solution and the notched-fatigue properties of some annealed brasses and condenser tube alloys with a rotating-beam fatigue tester. The effects of aluminum contents on the fatigue properties of aluminum brass were also studied with a Schenck's plane-bending fatigue tester. The relations between the fatigue strength at 10^8 cycles and zinc or zinc equivalents of the specimens are shown. The increases of zinc or zinc equivalents improve the mechanical properties and the unnotched-fatigue strength in air, but

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ALL—The Abstract Bulletin, Aluminium Laboratories, Ltd., P. O. Box 84, Kingston, Ontario, Canada.

ATS—Associated Technical Services Abstracts, Associated Technical Services, P. O. Box 271, East Orange, New Jersey.

AWWA—Journal, American Water Works Association, Amer. Water Works Assoc., Inc., 2 Park Ave., New York 16, New York.

BL—Current Technical Literature, Bell Telephone Laboratories, 463 West St., New York 14, New York.

BTR—Battelle Technical Review, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio.

CBEC—Centre Belge d'Etude de la Corrosion (CEBELCOR), 21 rue des Drapiers, Brussels, Belgium.

CDCT—Boletin, Centro de Documentacion Cientifica y Tecnica, Plaza de la Ciudadela 6, Mexico 1, D. F.

EL—Electroplating and Metal Finishing, 85 Udney Park Road, Teddington, Middlesex, England.

HB—Translations, Henry Brutcher, P. O. Box 157, Altadena, California.

IIM—Transactions of The Indian Institute of Metals, 31 Chowringhee Rd., Calcutta 16, India.

INCO—The International Nickel Co., Inc., 67 Wall Street, New York 5, New York.

JSPS—Japan Society for the Promotion of Science, Mr. Hayata Shigeno, Secretary, Committee of Preventing Corrosion, c/o Government Chemical Industrial Research Institute, 1 Chome Nakameguro, Meguro-Ku, Tokyo, Japan.

MA—Metallurgical Abstracts, Institute of Metals, 4 Grosvenor Gardens, London SW 1, England.

MR—Metals Review, American Society for Metals, 7301 Euclid Ave., Cleveland 3, Ohio.

NALCO—National Aluminate Corp., 6216 West 66th Place, Chicago 38, Illinois.

NSA—Nuclear Science Abstracts, United States Atomic Energy Commission, Technical Information Division, Oak Ridge, Tennessee.

OTS—Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C.

PDA—Prevention of Deterioration Abstracts, National Research Council, 2101 Constitution Ave., Washington 25, D. C.

PI—Pergamon Institute, 1404 New York Ave., N. W., Washington 5, D. C.; Oxford and London, England.

RPI—Review of Current Literature Relating to the Paint, Colour, Varnish and Allied Industries, Research Assoc. of British Paint, Colour and Varnish Manufacturers, Paint Research Station, Waldegrave Rd., Teddington, Middlesex, England.

SE—Stahl und Eisen, Verlag Stahleisen, m.b.H., Breite Strasse 27 (Schliessfach 2590), Duesseldorf, Germany.

TIME—Transactions of The Institute of Marine Engineers, The Memorial Bldg., 76 Mark Lane, London EC 3, England.

ZDA—ZDA Abstracts, Zinc Development Association, 34 Berkeley Square, London W 1, England.

excessive increments of these contents will not effect any improvement on the corrosion-fatigue and the notched-fatigue resistances. The corrosion-fatigue strength of copper-zinc binary alloy increases with zinc contents and then decreases with a rise over 20% of zinc contents. In the case of aluminum brass, Albrac alloy, Admiralty metal and arsenical Admiralty metal, the materials with rich zinc equivalent contents showed lower notched-fatigue and corrosion-fatigue strengths than the materials poor in zinc equivalent contents. These results show that the corrosion-fatigue strength is related to the notched-fatigue strength.—JSPS. 17635

2.3.7, 5.4.5

Methods Used in the Performance Testing of Paint. Pt. II. Adhesion. Pt. III. Hardness. W. V. Moore. Metal Finishing J., **3,** Nos. 30, 31; 241-242, 246; 285-288 (1957).

The problem of measuring the adhesion of a paint film in terms of the force per unit area required to remove the paint from the substrate is one of very great difficulty. Two very simple methods by which a comparison between the adhesion of two or more paints can be obtained are the cross-cut adhesion test and the adhesive tape test. A method of obtaining a quantitative measure of adhesion is the Gardner Adhesion Method. Each of these methods is described. The hardness of paints may be measured as the scratch hardness, rocker hardness and resistance to impact. Methods of measuring these types of hardness are briefly described.—RPI. 17907

2.3.7, 5.3.4, 6.4.2

Surface Treatment and Finishing of Light Metals. Part XIII. Plated Aluminum Tests, Corrosion, Applications. S. Wernick and R. Pinner. Metal Finishing, **55,** No. 6, 79-84 (1957) June.

Literature on adhesion of electroplates on aluminum is reviewed. Atmospheric exposure tests remain only reliable method for corrosion testing of electroplated aluminum; salt-spray and other accelerated tests are useful for setting minimum performance standards as long as their limitations are recognized. Corrosion resistance of aluminum plated with nickel, copper, nickel plus chromium and copper plus silver deposited by zincate process is discussed, with many references to work by F. Keller and W. G. Zelley. Test designed by Heussner, in which test samples are marked with V-shaped threading tool to form an X-pattern, is shown to be combined adhesion and corrosion test. Graph shows effect of thickness of nickel electrodeposit on corrosion rating of zinc, magnesium and aluminum alloys in industrial atmosphere. Photographs of specimens.—INCO. 16385

2.3.7, 3.4.9, 3.5.9

Humidity Testing of Protective Finishes. H. Melrose and R. E. J. Shave. Corrosion Prevention and Control, **5,** No. 5, 49-54 (1958) May.

Emphasizes importance of close control of humidity and of temperature, whether constant or cyclic to produce alternate condensation and re-evaporation of moisture on surface of finish. Attaining of very high relative humidity and thus dew point temperature close to dry bulb temperature during heating portion of cycle, involves very careful attention to design of test chamber and control of test air condition. Other factors considered are time period of successive cycles, size, shape and thermal mass of test specimen, thermal characteristics of test chamber, and distribution accuracy of air conditions within chamber working space.—INCO. 16709

2.3.9, 3.5.4, 8.4.5

Pre-Irradiation Tests on Uranium-Silicon Alloys. L. M. Howe and L. G. Bell. Atomic Energy of Canada Ltd. May, 1958, 41 pp. Available from The Scientific Document Distribution Office, Atomic Energy of Canada Ltd., Chalk River, Ontario, Canada.

Pre-irradiation tests of hardness, density, electrical resistivity and corrosion resistance as well as metallographic and X-ray examinations were undertaken on silicon-uranium core material, which had been co-extruded in Zircaloy-2, in order that the effect of irradiation on alloys in the epsilon range could be assessed. In addition, a study of the epsilonization of arc-melted material was undertaken in order to gain familiarity with the epsilonization process and to obtain information on the corrosion behavior of epsilonized material. Sheathed silicon-uranium samples in the epsilonized and de-epsilonized conditions were irradiated in the X-2 loop, with a water temperature of 275 C. The samples were examined after 250 Mwd/t and show no dimensional change. (auth)—NSA. 16500

2.3.9, 6.2.5, 3.4.3, 8.4.5

A Group Separation Method for Gamma Spectrometry of Complex Radionuclide Mixtures, with Special Application to Stainless Steel and Its Corrosion Products. K. Samsahl. Nuclear Chem. Lab., AB Atomenergi, Stockholm. Acta Chem. Scand., **12,** 1292-1296 (1958).

Short anion exchange columns coupled in series and saturated with chloride, citrate, and hydroxide ions have been used for simple and very rapid separations of common elements in water solutions as pretreatment for subsequent analysis with gamma spectrometry. A method was developed for the analysis of stainless steel, its corrosion products and other elements present as impurities in a water-cooled nuclear reactor. Practical examples are described in detail. With this method it seems possible to measure continuously water corrosion rates of individual or small groups of stainless steel elements. (auth).—NSA. 17655

2.3.9, 3.7.4, 3.8.4, 6.2.2

Electron Diffraction Studies of Crystal Structure of Oxides of Iron. A Pande (National Physical Lab., New Delhi, India). J. Sci. Ind. Research (India), **17B,** 205-208 (1958) June.

The crystal structure of oxide films formed on the surface of pure iron heated to different temperatures has been analyzed by the electron diffraction method. The effect of occluded hydrogen on the formation and orientation of oxides on the metal surfaces has also been studied by the same technique. The discrepancy in the value of the lattice parameters as determined by the electron diffraction and X-ray diffraction methods has been satisfactorily explained as primarily due to refraction of the electron beam reflected by the microcrystals of the oxides of iron. (auth)—NSA. 17529

2.3.9

Refinements in Radiographic Techniques. H. S. Wyckoff. Nondestructive Testing, **15,** No. 6, 336-339, 354 (1957) Nov.-Dec.

Laboratory testing procedures applied to checking of steel castings, welds, etc., found to be helpful in obtaining better results from well-established methods; three basic factors in obtaining radiographic quality work are proper use of equipment and processing methods; knowledge of fabrication procedures involved, and analysis of problem to obtain best technique for proper interpretation.—BL. 17973

2.3.9

Automatic Recording of the Fine Structure of X-Ray Absorption Spectra. K. Tsutumi, M. Sawada and A. Hayase. Norelco Reporter, **5,** No. 3, 55-57 (1958) May-June.

Fine structures of the X-ray K ab-

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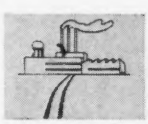

Chemical & Industrial Plant Piping

17a

sorption spectra of metallic cobalt and of iron in ferrous ammonium sulfate were recorded automatically using a Norelco Geiger Counter Spectrometer. Results are in agreement with those of the photographic method. Absorption coefficients of cobalt were measured. K emission spectra of copper and cobalt were recorded and resolving power was estimated. Tables, graphs.—INCO. 16638

2.3.9

A Method of Analyzing Wire Coatings on Copper Using Standard Metallographic Practices. D. Myers, Jr. Gen. Elec. Co. Wire and Wire Products, **33,** No. 6, 641-642, 704-705 (1958) June.

Describes new technique which permits examination of film on rectangular wire. Etching of samples of wire coated with Formvar-type enamels reveals individual layers in the coatings. Samples can be prepared in any rudimentarily equipped metallographic laboratory. When change in die design or arrangement is desired, technique is valuable in determining optimum design. Preparation, polishing and etching of sample are discussed.—INCO. 16545

2.3.9, 2.3.7, 1.3

The Measurement of Thickness. G. Keinath. National Bureau Standards Circular No. 585, iv + 79 pp. (1958).

A critical review (with 220 references) of the methods available for measuring the thickness of coatings, foils, metal panels, tubes, etc. The principles of the various techniques are outlined and reference is made to specific commercial instruments, with addresses of U. S. suppliers. Methods discussed include mechanical, optical, chemical, magnetic, electrical, X-ray and radioactive techniques.—RPI. 16558

2.4 Instrumentation

2.4.2, 3.2.2

Portable Magnetizer for Magnetic Crack Detection. J. W. Walley. Metropolitan-Vickers Gaz, **29,** No. 465, 118-121 (1958) April.

New type of hand-magnet, designed to provide easily adjusted magnetizing unit for rapid 'in situ' magnetic crack detection; two hinged pole-shoes are arranged to straddle line of suspected cracks; 'on-off' control of permanent magnet portion is obtained by movable soft-iron shunt; no power supply is needed.—BL. 16686

2.4.2, 4.2.1

Determining Corrosion Aggressiveness of the Atmosphere. N. D. Tomashov, G. K. Berukshits and A. A. Lokotilov. Corrosion Technology, **5,** 386-388 (1958) Dec.

Apparatus constructed at the Institute of Physical Chemistry of the U.S.S.R. Academy of Sciences and the I. V. Stalin Steel Institute. Used for study of a macro-model of a corrosion element which would operate in atmospheric conditions. 9 references.—RML. 17640

2.4.2

Sampling and Determination of Hydrogen in Steel Under Works Conditions. H. Zitter and H. Krainer. Arch. Eisenhüttenw., **29,** 401-410 (1958) July. (In German.)

Illustrated description of hydrogen determination equipment developed by Böhler for works application. Supporting data are given on applications of instrument to determination of hydrogen in low-alloy nickel, nickel chromium and nickel-chromium-molybdenum steels. —INCO. 17650

2.4.2

An Automatic Apparatus for Testing the Corrosive Action of Solutions. (In German.) Gerhard Wildbrett. Werkstoffe u. Korrosion, **9,** No. 8/9, 521-524 (1958).

The cleaning of foodstuff-industry equipment is considered. Machines for carrying out dip corrosion-tests automatically are described. The results of such tests are compared with manual operations. The automatic machine saves, in a set of tests, ~ 65% of the work.—MA. 17736

2.4.2, 2.3.9

A Radioactive Corrosion Probe. D. Anson. J. Inst. Fuel, **32,** 10-14 (1959) Jan.

Internally cooled steel probes are in general use for the estimation of corrosion rates of cooled surfaces exposed to flue gases. By using a radioactivated probe the metal lost during a test may be estimated by a count of the radioactivity of the probe washings. It may be possible to develop a probe on this principle to give a continuous indication of corrosivity. The author describes the design and performance of a simple prototype probe which demonstrated that accurate results should be possible using probe exposure times as short as 10 minutes, and counting times as 2 minutes with conventional counting equipment. With the design described, health hazards due to radioactivity are very slight provided that elementary precautions are taken to avoid direct handling of radioactive parts. 1 plate.—BTR. 17693

2.4.2, 5.8.1, 8.4.3, 8.9.3

Corrosion Rates Directly Measured by New Resistance Method. D. H. Stormont. Oil Gas J., **55,** No. 3, 85-87 (1957) Jan.

A portable d-c instrument, the "Corrosometer" is finding increased use for evaluating the effectiveness of chemical inhibitors in oil-field installations, pipelines, refineries and natural gasoline plants. Corrosion rates can be determined quickly and accurately by this electrical-resistance method which measures the loss of metal from a probe installed in the corrosive system. Resistance of the exposed specimen is measured by placing a second specimen of the same metal or alloy covered with a corrosion resistant coating in the probe and connecting in series to the first, making both part of a bridge circuit. The reference specimen retains its original cross section and resistance. Changes in the ratio of the two resistances, due to changes in the resistance of the exposed sample, are translated directly into units of corrosion by the meter circuit. Readings are essentially independent of the temperature and nature of the medium and are not affected by accumulation of scale or sludge. Basically, the meter circuit indicates the extent to which the metal is converted into nonmetal, and measurements are made without removing the probe from the corrosive environment. Probes used, which depend upon conditions of the systems and purpose of the study, include wire for long range testing or collection of control data and strip specimens which give maximum response to corrosion.

The insensitivity of Corrosometer to a-c pickup permits interference-free operation near pipelines and other underground structures. Resolution of the order of 0.5μ in. can be made on a strip specimen 4 mils thick. The instrument responds to the resistance of the exposed metal specimen as a whole, indicating changes as if the loss occurred uniformly over the entire surface. Thus, the data obtained are like the averages resulting from weight loss measurements.—PDA. 17890

2.4.3

Sources of Defects Located by Nondestructive Testing. C. E. Betz. Nondestructive Testing, **16,** No. 4, 312-332 (1958) July-Aug.

Consideration of methods used mainly for finding flaws or defects; role of inspector; defects are commonly classified on basis of progress of material from its raw state to finished form, namely, inherent, processing and service defects; examples relate to production of iron and steel pointing out sources of flaws within these groups.—BL. 18012

2.4.3, 2.3.9

Gamma Rays—X-ray Films—A New Way to Detect Corrosion. E. G. Pausohenwein and P. C. Trounce. Oil & Gas J., **56,** No. 51, 64-66 (1958) Dec. 22.

By combining gamma rays with X-ray film, corrosion can now be recorded and plotted with minute accuracy. Although prime use of this development is the detection of metal loss, it is proving useful in many other aspects of inspection work. These include welding inspection and the reexamination of internal mechanisms. Technique was developed by Sarnia Inspection Co., in cooperation with engineering department of Sarnia, Ontario refinery of Canadian Oil Companies, Ltd. Diagrams. —INCO. 17540

3. CHARACTERISTIC CORROSION PHENOMENA

3.5 Physical and Mechanical Effects

3.5.3

Corrosion-Erosion. (In French.) I. Ming-Feng and B. G. Rightmire. Navires, Ports et Chantiers, **8,** 757-759, 785 (1957) Oct.

Mechanism of corrosion; a curve represents the weight of lost metal as a function of number of effective cycles.—MR. 15374

3.5.3, 8.9.1

Supersonic Rain Erosion Testing of Missile Radomes. K. Barr and E. J. Steeger. Paper before Am. Rocket Soc., Spring Mtg., Washington, D. C., April 4-6, 1957. Jet Propulsion, **27,** No. 9, 1034-1037 (1957) Sept.

Data obtained from firing 20-mm and 57-mm cannons show that erosion damage of specimen, mounted on nose of projectile, is function of velocity, shape, material, water drop size and distance water drop must travel in flow field aft of shock. To obtain quantitative results for rain damage to total surface, full scale tests were run by mounting radomes on rocket sled and firing through simulated rainfall. Design and operation of rocket sled are given. SAE 4130 was chosen for all primary structural members. Type 321 inserts are used in each slipper to provide wearing surface that can be replaced after one or more runs. Operation at speeds of Mach 2 and above caused extreme wear of inserts. Flame patterns from insert burning

could be seen extending over 9 ft. behind slippers; flashes disappeared with entry of vehicle into rain range. Analysis of this leads to belief that test vehicle is actually running on molten layer of metal acting as a liquid lubricant.—INCO. 16125

3.5.4, 5.4.5

How Radiation Affects Six Organic Coatings. Lloyd A. Horrocks. Materials in Design Engineering, **47,** 120-123 (1958) Jan.

The effects of radiation on phenolic, epoxy, and fluorinated vinyl coatings, nitrocellulose lacquers, and alkyd and silicone-alkyd enamels were evaluated. —BTR. 15456

3.5.8, 6.2.3

Corrosion Fatigue of Steel Wire. (In Japanese.) Endo Kichiro, Hiroyasu Huji and Shunji Omori. Trans. Soc. Mech. Engrs., Japan, **23,** 484-488 (1957) July.

Corrosion fatigue of 0.75 percent carbon steel, annealed and cold worked, in relation to residual stress.—MR. 14666

3.5.8, 3.7.4, 6.2.3

Stress Condition for Brittle Fracture of Mild Steel. T. Yokobori, H. Hamamoto and A. Otsuka. Nature, **181,** No. 4625, 1719-1720 (1958) June 21.

Description of tensile tests on specimens of normal shape and torsion tests on thin-walled hollow cylinders carried out at liquid-air temperature. Theories proposed for fatigue fracture are discussed. It is interesting that in torsion test the main crack forms a spiral in a direction at 49-52° to axis of specimen, whereas in tension test surface fracture conforms macroscopically to a cross-section of specimen without any tendency to necking. These facts, together with relation, suggest that criterion of fracture may not invariably correspond to macroscopic geometry of pattern of fracture, as has usually been assumed. Tables, photos. (auth)—INCO. 16673

3.5.8, 3.2.2

Watch Out for Stress Corrosion in Metals. E. H. Phelps. Product Eng., **29,** 56-58 (1958) August 4.

Stress in certain chemical environments leads to intergranular and transgranular corrosion. Design principles to avoid this corrosion.—MR. 16706

3.7 Metallurgical Effects

3.7.2, 3.4.8, 6.2.2

Effect of Titanium, Vanadium, Tungsten and Zirconium on the Sulphurization-Resistant Property of Cast Iron at High Temperatures. (In English.) Masakazu Shiozawa and Hiroshi Nakai. Waseda University, Castings Research Laboratory, Report No. 8, 13-15 (1957) Nov.

Alloy specimens were exposed to hydrogen sulfide at 900 C for 10 hr.—MR. 16334

3.7.2

The Influence of Alloying Additions on the Processes of Oxidation of Metals During Melting and Refining. (In Polish.) K. Kurski. Rudy i Metale Niezelazne, **2,** No. 1, 27-30 (1957).

Kurski studied the oxidation of lead containing varying amounts of aluminum, tin, zinc, cadmium, sodium, calcium and copper taken separately, as well as tin plus aluminum and cadmium plus zinc, comparing the oxidation rate of the alloys with that of the base metal. It was found that alloying elements can minimize the oxidation loss. Additions of 0.01 percent aluminum to zinc can save ~10 kg zinc/ton. Additions of small amounts of zinc to molten cadmium reduces the oxidation loss of cadmium. The theory of such protection is given.—MA. 15804

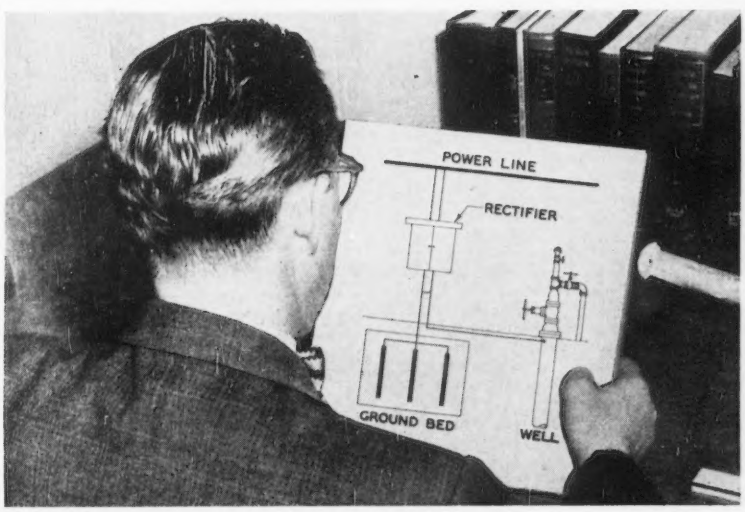
3.7.3, 3.5.6

Gas Porosity and Sources of Hydrogen in the Metal-Arc Welding of Light Alloys. W. G. Hull and D. F. Adams. Brit. Welding J., **5,** No. 6, 282-290 (1958) June.

Experimental work, using electrodes having aluminum-10 silicon core, shows that hydrogen may be derived from number of sources within electrode including free moisture, water of crystallization and corrosion product at core-wire surface, and that each individually can contribute towards weld metal porosity. Although moisture can be removed by baking, any significant improvement in soundness hinges on development of fluxes which are free from water of crystallization and also on prevention of core-wire corrosion by surface protection or control of storage conditions. Among photomicrographs is one showing unsoundness produced in weld metal from bare core wire corroded by coating during storage. Tables.—INCO. 16535

3.7.4.

On an Increased Reactivity at the Grain Boundaries of Solid Solutions. (In German.) L. Graf and H. Klatte. Z. Phys. Chem., Frankfurt, **10,** 306-309 (1957) March.—BL. 14632



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3.7.3, 6.3.20, 8.4.5

Brazing of Zircaloy 3 for Temperature Instrumented Probes. J. M. Gerken. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-JMG-9, May 1, 1957, 11 pp. Available from Office of Technical Services, Washington, D. C.

None of the brazing materials tested in this program was considered satisfactory for brazing Zircaloy for service in high temperature pressurized water. The titanium plus 1 percent tin and titanium plus 2 percent tin brazing materials produced joints having good strength and ductility but both disintegrated completely in corrosion test. The copper-palladium-idium alloy, which apparently corroded the least of the alloys tested, was extremely brittle and therefore was considered unsuitable from a mechanical standpoint for many types of joints. (auth).—NSA. 15286

3.7.3, 6.2.5

Distribution of Carbon in the Heat-Affected Zones of Welds in Austenitic 18-8 Steel. N. V. Ul'yanova and B. V. Sagalovich. Metallovedenie i Obrabotka Metallov, No. 7, 2-7 (1957) July. Translation available from: Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Autoradiographic study of carbon distribution in heat-affected zone of argon-arc welds in austenitic 18-8 steels immediately after welding and after various heat treatments, to throw light on structure of metal near the fusion line, known to be susceptible to damage in service even if titanium or columbium stabilized. Correlation of observations with corrosion in fuming and boiling 65 percent nitric acid (knife-line attack). Best heat treatment for avoidance of weld decay or corrosion in service. 5 figures.—HB. 16381

3.7.3, 6.2.5

Results of Corrosion Tests of Type 347 Stainless Steel Weldments in Solutions Supplied by the Chemical Technology Division. C. C. Vreeland. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-53-1-256, Jan. 24, 1953 (Declassified April 2, 1957), 8 pp. Available from Office of Technical Services, Washington, D. C.

Stainless steel weldments for the Thorex Pilot Plant were corrosion tested in Thorex AW stream and 3-20 decontamination solutions. Uniform weight losses were observed for 3 specimens which were examined metallographically and with a low-power microscope. No excessive attack was noted on the weld metal or base metal.—NSA. 16373

3.7.3

Chlorine Additions for High-Quality Inert-Gas Metal-Arc Welding of Aluminum Alloys. M. B. Kasen and A. R. Pfluger. Welding J., Supplement, **37,** No. 6, 269s-276s (1958) June.

Contamination on the surface of aluminum filler wire is often the cause of poor quality inert-gas metal-arc welds. This paper deals with additions of chlorine to the arc area to eliminate unsoundness from such contamination. It covers multipass flat-position welds using aluminum-magnesium plate and filler wire. A study was made in which the liberation of contamination from the filler-wire surface was simulated by addition of hydrogen, propane, water vapor or oxygen to the arc area through the contact tube of the welding torch. The quantity of each of the contaminating agents necessary to produce a weld of very poor quality was determined. Chlorine added to the arc area in the range of 3 to 20 cu cm/min was found to counteract effectively the deleterious effects of each of these contaminators. Further tests with purposely contaminated filler wires resulted in an equally marked benefit. A small quantity of chloride permitted radiographically sound welds to be made with mill-run wire and with wire contaminated by a hydrated oxide. Indications were obtained that chlorine additions can also overcome the effect of contamination which naturally occurs on commercial welding wire of the aluminum-magnesium type, thus providing insurance against unsound welds resulting from wire which has been improperly cleaned or which has become recontaminated during storage or use. A chlorine flow of 10 cu cm/min added to the arc area through the contact tube of the welding torch is suggested as optimum. Recommendations are given on methods to obtain this flow rate and on measures which must be taken to overcome corrosion problems created by the chlorine. It is concluded that welding with the minute quantities of chlorine described in this paper can be safely undertaken in any welding area adequately ventilated for normal welding. (auth)—ALL. 16733

3.8 Miscellaneous Principles

3.8.2, 3.4.8, 6.3.14

Research on the Electrochemical Behaviour of Tin. Pt. II. Solutions Containing Fluoride Ions. U. Bertocci and G. Serravalle. Metallurgia Italiana, **49,** No. 2, 95-98 (1957).

Bertocci and Serravalle studied tin electrodes in F^- and Cl^-—F^- solutions, with 0.5M — Sn^{II}, at 25-65 C. In neutral solutions the electrode reaction is: $SnF_3^- \rightarrow Sn + 3F^-$; with 4-6M-ammonium fluoride there is only a low overvoltage at both anode and cathode, but at 3M-NH_4F the anodic polarization becomes higher. The addition of small quantities of hydrochloric acid to neutral solutions has but little effect on the overvoltage.—MA. 14942

3.8.2

Corrosion as an Electrochemical process. (In German.) F. W. Hill. Praktische Chemie, **8,** 238-243 (1957) August.

Form and extent of corrosion of steel construction when submitted to uncontrolled electrochemical reactions.—MR. 15300

3.8.2, 6.2.2

Action of Alternating Current on Corrosion Process of Iron. Yutaka Torigoe. J. Electrochem. Soc., Japan, **28,** E-30-E-33 (1958).

The effect of alternating current on the local cells on iron in a given corrosive environment is determined by the impressed potential, the polarization characteristics of the local anode and cathode and the magnitude of alternating current applied.—MR. 16648

3.8.2, 3.6.5, 3.6.8

Electrochemical Principles of Metal Protection in Strong Oxidizers. (In Russian.) V. P. Batrakov. Doklady Akademii Nauk SSSR, **117,** No. 2, 252-254 (1957) Nov. 11.

Recommends decreasing the reduction-oxidation potential of the medium, increasing the overvoltage of the anodic process, or increasing the overvoltage of the cathodic process.—BTR. 15528

3.8.2, 6.2.2, 6.2.3

Dissolution of Metals—Especially Iron and Mild Steel in Acid. (In Norwegian.) Tor Hurlen. Teknisk Ukeblad, **105,** Nos. 5, 6; 95-101, 119-122 (1958) Jan. 30, Feb. 6.

A study of hydrogen-developing corrosion types, based on electrochemical theory. Kinetics of the electrode reactions. Anodic solution of iron. Cathodic development of pH.—BTR. 16530

3.8.3, 6.4.4

On Negative Difference Effect in the Anodic Polarization of Magnesium. (In Russian.) D. V. Kokoulina and B. N. Kabanov. Doklady Akad. Nauk SSSR, **112,** No. 4, 692-695 (1957). 14670

3.8.4, 4.3.3, 3.7.2

A Microscopic Study of Oxide Films Formed on Chrome-Iron Containing Approximately 1.25% Copper Exposed to 0.17 M Uranyl Sulfate Solution at 250 C for 12 Weeks. T. E. Willmarth and F. D. McNeer. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-52-1-181, Jan. 23, 1952 (Declassified Feb. 14, 1957), 17 pp. Available from Office of Technical Services, Washington, D. C.

A tube section of No. 443 chrome-iron with 1.25 per cent copper addition was immersed for 12 weeks in a 0.17M uranyl sulfate solution at 250 C. No passivation pretreatment was used. The oxide film formed in contact with the metal substrate appeared to meet the conditions essential for a protective film. The presence of Cu^{2+} in the metal alloys assisted the formation of the film. The addition of copper to other steels, preferably of the 300 series, also produced a material relatively free from corrosion attacks in uranyl sulfate solution.—NSA. 16359

3.8.4, 4.2.1

Chemical Proof of Hydrogen Peroxide Formation in the Atmospheric Corrosion of Metals. (In Russian.) I. L. Roikh. Zhur. Fiz. Khim., **32,** No. 5, 1136-1142 (1958) May.

The application of direct chemical methods has proved the formation and the emission of hydrogen peroxide in atmospheric corrosion of both the electronegative and the electropositive metals. This result was demonstrated by experiments with 12 various reactions on 19 metals.—BTR. 16594

3.8.4, 3.8.2

Phase Diagram of the System Iron Compounds—Aqueous Medium in the Coordinates φ redox-pH. (In Russian.) O. N. Lapteva. J. Applied Chem., USSR (Zhur. Priklad. Khim.), **31,** No. 9, 1300-1303 (1958).

The equilibrium $Fe^{++} \rightleftharpoons Fe^{+++} + e$, $Fe^{++} + 3H_2O \rightleftharpoons Fe(OH)_3 + 3H^+ + e$, $3Fe^{++} + 4H_2O \rightleftharpoons Fe_3O_4 + 8H^+ + 2e$, $Fe_3O_4 + OH^- + 4H_2O \rightleftharpoons 3Fe(OH)_3 + e$, and $3Fe + 8OH \rightleftharpoons Fe_3O_4 + 4H_2O + 8e$ are characterized. 2 tables, 2 figures, 26 references.—ATS. 16562

3.8.4, 6.3.15

On Oxidation Mechanisms of Metals and Alloys. J. Markali. Research, **10,** 367-369 (1957) Sept.

A study of titanium and titanium alloys.—BTR. 15366

3.8.4, 4.3.7, 3.5.9, 6.3.10

The Oxidation of Nickel in Sulfur Dioxide at High Temperatures. V. V. Ipat'ev and D. V. Zheltukhin. Zhurnal Prikladnoi Khimii (J. Applied Chem.), U.S.S.R., **30,** No. 9, 1281-1286 (1957). Available from Associated Technical Services, Inc., P. O. Box 271, East Orange, New Jersey.

Above reaction, $3\ Ni + SO_2 = 2NiO + NiS$, is studied between 600 and 800 C. 3 tables, 6 figures, 6 references.—ATS. 15336

3.8.4, 4.7

Dynamic Solution-Rate Studies of Solid Metals in Liquid Metals. A. G. Ward and J. W. Taylor. J. Inst. Metals, **86,** No. 1, 36-42 (1957-1958).

A study was made of the kinetics of solution of solid copper in liquid lead and bismuth under dynamic conditions in the temperature range 360-460 C. and the solution process is shown to conform to an expression of the form: $n_t = n_o [1 - \exp[-Kt(S/V)]]$, where n_t = solute concentration at time t, n_o = saturation concentration at a fixed temperature, K = solution rate constant, S = surface area of solid exposed, V = volume of solvent. This expression is similar to that which applies in nominally static tests. From the variation of K with temperature and velocity in the two systems studied, it is concluded that the effective rate-controlling step in the dissolution process, under dynamic conditions, is the rate at which solute atoms are transferred from a saturated layer at the solid/liquid interface into the bulk of the liquid by turbulent and convective flow. The validity of a general relationship between the reduced concentration (n_t/n_x) and the reduced time (t/t_x) is established for dissolution at any temperature and under any dynamic condition; n_x is any fraction, x, of the saturation concentration n_o, and t_x is the time for the concentration to reach n_x. The use of this general relationship in interpolating complete concentration/time curves from limited experimental data is outlined. (auth)—MA. 15160

4. CORROSIVE ENVIRONMENTS

4.3 CHEMICALS, INORGANIC

4.3.2, 6.3.16

The Dissolution of Tungsten in Mixtures of Hydrofluoric and Nitric Acids. D. Robbins. Metallurgia, **55,** 257-259 (1957) May.

Metal most readily dissolves in a mixture containing about 40 per cent by volume concentrated nitric acid and 60 per cent concentrated hydrofluoric acid. —BTR. 14800

4.3.3, 7.6.5

Knitted Wire Mesh Knocks Out Caustic Mist. T. W. Wett and H. V. Coats, Jr. Frontier Chemical Co. Chem. Processing, **20,** No. 9, 176-177, 180 (1957) Sept.

Entrained liquid in vapor stream from sodium hydroxide evaporator at Frontier Chemical Co., was destroying large quantity of 30" steel vapor line. Solution was installation of Metex knitted wire mesh mist eliminator in caustic evaporator. Mist eliminator is essentially a 6-inch pad of uniform density made of layers of knitted nickel wire. Structure has maze of unaligned, asymmetrical openings, an unusually high free volume (97-98 per cent) and impingement target area 100 sq ft per cu ft.—INCO. 16419

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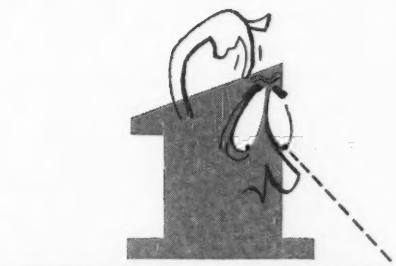

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4.3.5, 5.8.1, 6.2.5

Inhibition of Fluoride-Catalyzed Corrosion of Stainless Steels. R. L. Moore and R. A. Watts, Jr. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-33075, September 16, 1954 (Declassified April 4, 1957), 6 pp. Available from Office of Technical Services, Washington, D. C.

A series of experiments was conducted to demonstrate the magnitude of fluoride corrosive behavior and to test a number of substances as possible corrosive inhibitors.—NSA. 16250

4.3.5

Nonmetallic Material Compatibility with Liquid Fluorine. H. G. Price, Jr. and H. W. Douglass. Lewis Flight Propulsion Lab. National Advisory Cttee. for Aeronautics, Research Memo E57-G18, October 2, 1957, 7 pp.

Static tests were made on compatibility of liquid fluorine with several nonmetallic materials at —320 F and at pressures of 0 and 1500 pounds per square inch gage. Polytrifluorochloroethylene and N-43, did not react with liquid fluorine at atmospheric pressure of 1500 pounds per square inch gage under static conditions, but they did react when injected into liquid fluorine at 1500 pounds per square inch gage; they also reacted with gaseous fluorine at 1500 pounds per square inch gage. While water did not react with liquid fluorine at 1500 pounds per square inch gage, it is known to react violently with fluorine under other conditions. Pipe-thread lubricant Q-seal did not react with liquid fluorine, but did react with gaseous fluorine at 1500 pounds per square inch gage. Of solids, ruby (aluminum oxide) and Teflon did not react under the test conditions. Tables.—INCO. 16241

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4.6 Water and Steam

4.6.2, 7.4.1, 7.6.4

Protection Against Corrosion in Industrial Plants Using Steam. (In French.) R. Malicet. Corrosion et Anticorrosion, **5,** 174-184 (1957) June.

Types of corrosion prevalent; various types of attack to which equipment is subjected; boilers, superheaters and piping; factors influencing the corrosion mechanism. (To be continued.)—MR. 14730

4.6.2, 3.8.4

Oxidation of Steels in Superheated Steam. (In Czech.) Pavel Gröbner and Zdenek Bret. Hutnicke Listy, **12,** No. 2, 125-131 (1957).

Experiments demonstrate that it is possible to apply Wagner theory on iron and steel oxidation in superheated steam with sufficient exactness for practical purposes. 23 references.—MR. 15814

4.6.2, 6.3.6, 7.6.4, 3.5.3

The Examination of a Case of Corrosion in a Reheater of a Power-Station Boiler. J. Schoofs. Bull. Centre Belge Etudes Document. Eaux. No. 83, 436-439 (1957).

Serious corrosion of tubes of 70:30 brass carrying water entering at a temperature of 125 C and heated to 285 C occurred mainly where the direction of the flow changed abruptly. Erosion was the main cause of failure. The internal surfaces of the tubes were covered with a very thin brown film of iron oxide beneath which black copper oxide and a dezincified layer were also found. This protective layer was absent in corroded regions. The design of the reheater was such that overheating and local boiling took place with a disruptive effect on the protective film, particularly at the exit and entry where turbulence was greatest. Annealed "Perennic 10" instead of 70:30 brass, belling out of the ends of the tubes and the insertion of a baffle plate of "Super Perennic 30" to prevent over-heating are recommended. —MA. 16327

4.6.2, 6.6.6, 3.5.9

Tests for Evaluating Resistance of Porcelain Enamel to Steam Condensate Attack. J. A. Schiefferle. Paper before PEI Shop Practice Forum, 18th Annual Mtg. Metal Products Mfg., **14,** No. 9, 28-30, 32 (1957) Sept.

With introduction to major appliance field of combination washer-dryer, there has arisen a new problem of steam condensate corrosion of porcelain enamel. Survey of test procedures used to determine corrosion is given. Some investigators reported that angle of panel is very important. If panels are in a position that droplets formed are retained on panel, a selective etching will be encountered. Temperature of test specimens was reported as being very important as attack diminishes very rapidly below 190 F. Above 200 F very little condensate forms on panel. This seems to point out importance of controlling panel temperature. Several procedures take into consideration rate of amount of steam condensate formed as means of controlling amount of attack. Photos. —INCO. 16305

4.6.2, 5.7.9, 3.2.3

Theory and Practice of Boiler Water Treatment as a Means of Eliminating Corrosion and Scale. E. Quaade-Holm. Corrosion Technology, **5,** 213-214, 237 (1958) July.

An organic compound of high molecular weight not only eliminates scale-forming compounds, but removes old scale. The scale is physically removed rather than chemically decomposed.—MR. 16596

4.6.4

Treat Your Cooling Water Right. R. G. Dalbke and J. M. Masterson. Dearborn Chem. Co. Power Eng., **61,** No. 7, 56-59 (1957) July.

Discussion of scale formation, corrosion, biological growth and wood deterioration encountered in operation of cooling water systems and controlling treatments.—INCO. 14614

4.6.4, 6.4.3, 8.4.5

The Corrosion of Beryllium in Simulated Cooling Water for the Proposed Development Reactor (Interim Report for Period September 1946 to December 1948). James L. English. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-298, March 17, 1949 (Declassified Feb. 7, 1957), 129 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion studies on vacuum cast and extruded beryllium in simulated cooling water depend on metal purity, metallurgical treatment of metal, quality of cooling water, and the effect of solution velocity and stagnant areas in the water. The presence of oxygen tended to reduce corrosion for beryllium but increased it in bimetallic couples.—NSA. 14958

4.6.5, 6.3.20, 6.3.13, 6.3.5

Corrosion of Zirconium, Exploratory Tests on Tantalum and Niobium Alloys in Distilled Water. R. A. U. Huddle and C. F. Britton. Gt. Brit. Atomic Energy Research Establishment, Harwell, England, M/M-41 (Rpt: Unclassified), Feb., 1952 (Declassified April 2, 1957), 4 pp.

Corrosion of zirconium, niobium, and tantalum alloys in distilled water at 200 and 300 C is discussed.—NSA. 14698

4.6.11, 6.3.6

Corrosion Resistance of Copper and Copper-Alloys Tubes Used to Carry Sea Water. (In German.) K. Eichhorn. Werkstoffe u. Korrosion, **8,** 453-456 (1957) August/September.

Review of the corrosion resistance of copper and copper-base alloy tubes used in the sea-water systems of ships. Notes are given on corrosion-resistance of copper: failure with increase in rates of water-flow due to erosion-corrosion. Satisfactory use is disclosed of nickel-copper-iron alloy (5 nickel, 1 iron) and aluminum brass (2 aluminum) at these higher rates.—INCO. 14940

4.7 Molten Metals and Fused Compounds

4.7

Coating Steel with Molten Zinc. S. Socha. (In Polish with short English Summary.) Prace Inst. Minist. Hutn., **9**, No. 6, 269-279 (1957).

The paper discusses how the solubility of steel in molten zinc is affected by small amounts of metals, such as lead, cadmium and tin, in the zinc. Figures.—ZDA. 16316

4.7, 3.8.4, 6.3.21

The Dissolution of Germanium by Molten Indium. B. Goldstein. RCA Review, **18**, 213-220 (1957) June.

Experiments were performed on dissolution as a function of crystal axis, temperature, and germanium concentration of the solvent. Plots of germanium dissolution vs. time are generally characterized by two regions indicating initial and final rates.—BTR. 14620

4.7, 5.8.2, 5.3.4, 6.2.4

The Corrosion of 2¼% Chromium-1% Molybdenum Steel by Liquid Bismuth. G. W. Horsley and J. T. Maskrey. J. Iron and Steel Inst., **189**, Pt. 2, 139-148 (1958) June.

Dynamic corrosion of 2¼ chromium-1 molybdenum silicon-killed steel superheater tubing by liquid bismuth was studied in thermal convection loops working with maximum temperatures up to 625 C, temperature gradients up to 150 C and fluid velocities of 3-4 mm/s. Under these conditions rate at which steel is corroded by pure bismuth is at least 0.65 in./yr. Effect of adding 250-500 ppm zirconium to bismuth as an inhibitor on corrosion of steel was studied. This inhibitor plates out on the steel mainly as a film of zirconium nitride and temporarily prevents attack by bismuth, reducing corrosion rate to about 0.17 in./yr. The film however, occasionally spalls off and is not reformed; under this condition intergranular corrosion of underlying steel proceeds rapidly. If steel is heavily nitrided (1 per cent nitrogen) its rate of corrosion by inhibited bismuth is reduced to a maximum of 0.010 in./yr., nitride film reforms if broken and intergranular penetration is prevented. Effect of pile radiation in BEPO on corrosion of steel by inhibited bismuth was also studied. It has no beneficial effect and may even considerably reduce corrosion resistance of steel and efficiency of inhibitor. Diagrams, photomicrograph, graphs.—INCO. 16505

4.7, 6.3.15

The Attack of Liquid Zinc on Titanium. (In German.) K. Ruttewit and E. Eichmeyer. Metall, **11**, 659-662 (1957) August.

Attack of liquid zinc on titanium is linearly proportional to time and temperature. Titanium loss is appreciable and dissolves completely after a few hours at 600 C.—BTR. 14802

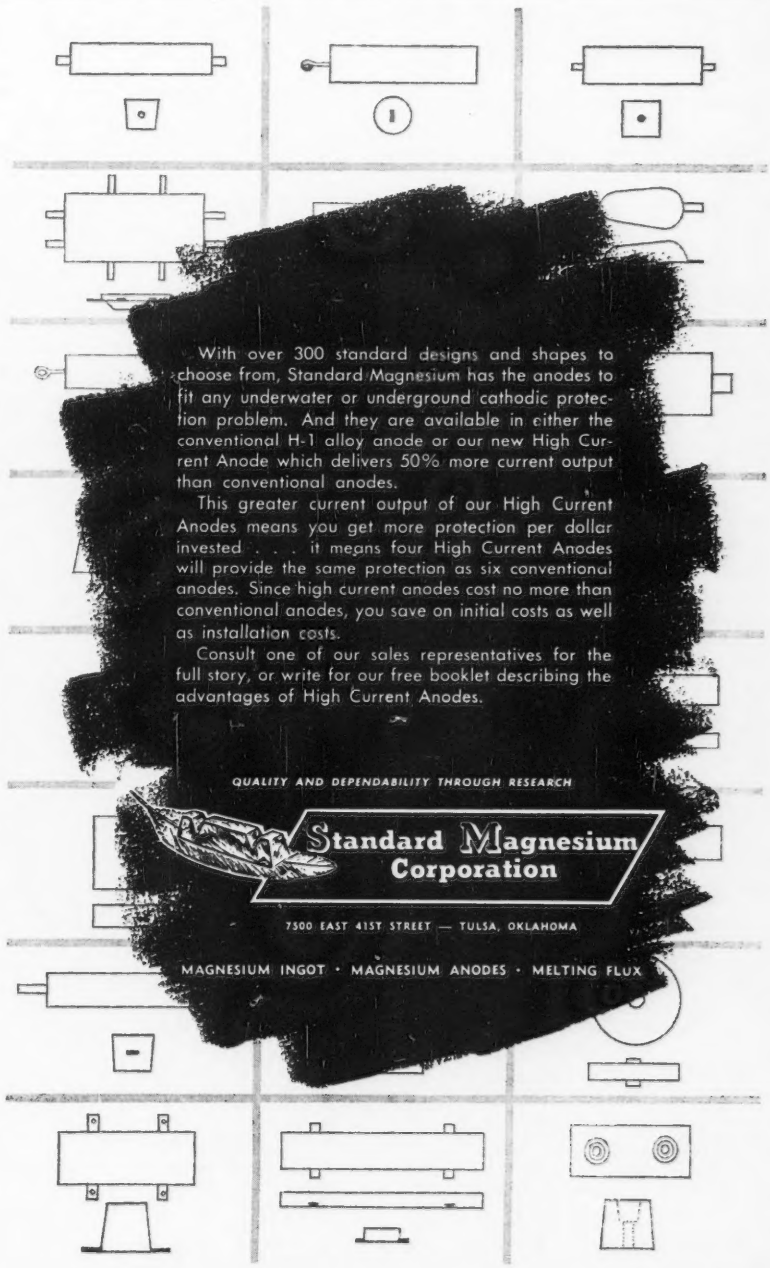
4.7, 7.6.4, 5.11

The Design of a Liquid Metal Heated Bayonet Tube Steam Generator. F. Boni. Griscom-Russell Co. J. Am. Soc. Naval Engrs., **70**, No. 2, 231-243 (1958) May.

Problems encountered in design of liquid metal heated steam generators are considered. Large temperature differences result from low heat capacity of liquid metals, high operating temperature range of liquid metals and low critical temperature of water. Sodium or liquid metal and water interleakage create such problems as liberation of hydrogen resulting in pressure rise, corrosion on liquid metal side by sodium or potassium oxides on hot containing elements, and corrosion and caustic embrittlement on water side by hydroxide solution at temperature. Unit under consideration includes evaporator, superheater and steam drum. Stress analyses, both theoretical and experimental, are discussed. Materials of construction are reviewed. Austenitic stainless steels, while resistant to sodium, are susceptible to stress corrosion cracking on water side. Conventional boiler materials, e.g., ASME Specification SA-212, can be used on water side of evaporator because oxygen corrosion problem encountered is well understood and can be controlled. On steam side of superheater 2¼ chromium-1 molybdenum steel is selected.—INCO. 16437

The Wetting of Solids by Liquid Metals. B. R. T. Frost. A.E.R.E., Harwell. Atomics, **8**, 387-390 (1957) October.

The fundamentals of wetting are reviewed, and methods of measuring the wetting of metals by liquid sodium, bismuth and lead are discussed. Reactor problems concerned with wetting properties are summarized. These are concerned with corrosion, slurries or suspensions, and thermal and electrical conductivity.—NSA. 15253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5. PREVENTIVE MEASURES

5.2 Cathodic Protection

5.2.1

Low-Cost Corrosion Control with Minimum Downtime. G. Weyermüller and W. V. Bacon. Food Machinery and Chem. Corp. Chem. Processing, **20,** No. 8, 108-109 (1957) August.

Water-purification equipment, consisting of 2 Accelerator units and chemical holding tank with mixer and chlorine diffuser, was installed at Westvaco Chlor-Alkali, with considerable amount of steel submerged. No. 17ST4 aluminum anodes were installed horizontally and vertically, and a 12½ amp, 34-volt rectifier was used for each of the 3 vessels. No corrosion was observed after 3 years. Only 4 hours downtime is required on cathodic protection system to change all anodes; this is done once a year.—INCO. 16394

5.2.1, 5.4.5

How Cathodic Protection Affects Paints and Coatings. H. S. Preiser and F. E. Cook. J. Bureau of Ships, **6,** 15-18 (1957) Sept.; Modern Plastics, **35,** No. 3, 175 (1958).

The effects of controlled potential on underwater coatings were determined by a series of tests. Results of these tests on vinyl, Neoprene, epoxy, polyethylene, porcelain enamel and other coatings are presented.—RPI. 16236

5.2.1, 5.4.5, 4.6.11

Fight Against Sea Corrosion of Steel with Cathodic Protection and With Paint. (In French.) M. Fonteyn. Centre Belge d'Etude et de Documentation des Eaux, Bulletin No. 40, 101-102 (1958).

Simultaneous action by both means produces practically a total prevention against sea corrosion.—BTR. 16486

5.2.2

The Magnesium Anode for Cathodic Protection. (In Norwegian.) T. Noess and J. B. Ostby. Tekn. Ukeblad, **105,** 403-408 (1958) May 1.

The theory underlying the use of magnesium for cathodic protection and the application of magnesium and magnesium alloy electrodes for this purpose are discussed.—ALL. 16616

5.2.2, 5.2.3

Cathodic Protection for the Chemical Industry. C. L. Wilson. Chem. and Process Eng., **38,** No. 10, 394-395 (1957).

The relative merits of the impressed-current and the sacrificial-anode systems are described for condensers, storage tanks, pumps, etc.; the anode materials for each system are compared.—MA. 16352

5.2.2, 8.9.5, 7.6.4

Cathodic Protection in Israel. Pt. 2. On Sea and in the Home. D. Spector. Corrosion Technology, **4,** 306-309 (1957) Sept.

Cathodic protection of Israel's fishing fleet and household hot water boilers by means of magnesium anodes.—MR. 16296

5.2.2, 5.2.3

Underground Corrosion and Cathodic Protection of Gas Pipelines. J. S. Gerrard. Gas J., **293,** 503-506 (1958) March 5.

Nature of the attack; methods of corrosion reduction; magnesium anodes and impressed current systems.—MR. 16484

5.2.4, 4.6.11

Cathodic Protection of Steel in Salt Water. National Bureau of Standards, Technical News Bulletin, **42,** 83-84 (1958) May.

The National Bureau of Standards has completed a study of the electrical potential needed to protect steel cathodically from corrosion in salt water.—BTR. 16620

5.4 Non-Metallic Coatings and Paints

5.4.2, 6.4.2

Enamels for the Protection of Aluminum Cans for Uranium Slugs. John Schultz, Harlan P. Tripp, Burnham W. King and Winston H. Duckworth. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1034, Aug. 31, 1955 (Declassified Feb. 12, 1957), 22 pp. Available from Office of Technical Services, Washington, D. C.

Enamels for 2S aluminum were developed which exhibited corrosion rates of about 1/75 that of bare aluminum when exposed to boiling alkaline water for 100 hr. These enamels were based on the sodium oxide-lead monoxide-silicon dioxide eutectic composition with substitutions of a total of 10 to 15 w/o of titanium dioxide, zirconium dioxide and alkaline earth oxides for lead monoxide and sodium oxide. About 1 w/o nickel monoxide was beneficial for promoting adherence. Enamels having the lowest corrosion rates all contained small amounts of lithium oxide. Best results were obtained when 2S aluminum was used as the base metal for enameling. Other aluminum alloys, EC metal and 63S, also enameled satisfactorily. However, 24S alloy did not survive the firing operation necessary to fuse the enamel to the metal. The presence of pinholes and fractures in the enamel resulted in pitting of the exposed aluminum at rates two to three times greater than that of completely bare aluminum under the same conditions. Therefore, defect-free enameling would be required to obtain suitable protection. (auth)—NSA. 16329

5.4.3, 4.3.2

Rubber-Lined and Neoprene-Lined Tanks Resist Corrosive Acid Solutions. G. Weyermuller and L. Painter. Uranium Reduction Co. Chem. Processing, **21,** No. 6, 141 (1958) June.

One of largest installations of rubber-lined and neoprene-lined tanks in the country is giving excellent service in handling corrosive leaching and ion exchange solutions used in purification of uranium. Tanks are still in excellent condition after 1½ years of continuous use handling solutions containing sulfuric acid and nitric acid.—INCO. 16690

5.4.2, 6.2.2

Processes Occurring During Enameling of Cast Iron. D. Kamran. Giesserei, **44,** 705-713 (1957) Nov. 21.

Occurrence of scale, rate of oxidation of cast iron in different atmospheres and after treating with enameling compounds, effect of addition elements on ease of enameling of cast iron and adhesion of enamel to cast irons, including spheroidal graphite iron, are discussed.—INCO. 15319

5.4.2, 4.3.3, 6.4.2

These Aluminum Enamels Resist Corrosion by Boiling Alkaline Water. B. W. King, W. H. Duckworth, John Schultz

No other supplier offers you as many materials or as much practical experience in corrosion control. Federated's Corrosion Advisory Service can recommend the best for you from among GALVANIC ANODES, magnesium and zinc; LEAD SHEET, PIPE, and FITTINGS; ZINC and ZINC ALLOYS for galvanizing; ZINC DUST; COPPER and ALLOYS; and PLATING MATERIALS, including copper, lead, cadmium, zinc, and silver anodes; nickel salts and addition agents for plating baths. One of Federated's 22 sales offices is near you. Don't hesitate, call us with your corrosion problems . . . no obligation, of course. Federated Metals Division, 120 Broadway, New York 5. In Canada: Federated Metals Canada, Ltd., Toronto and Montreal.

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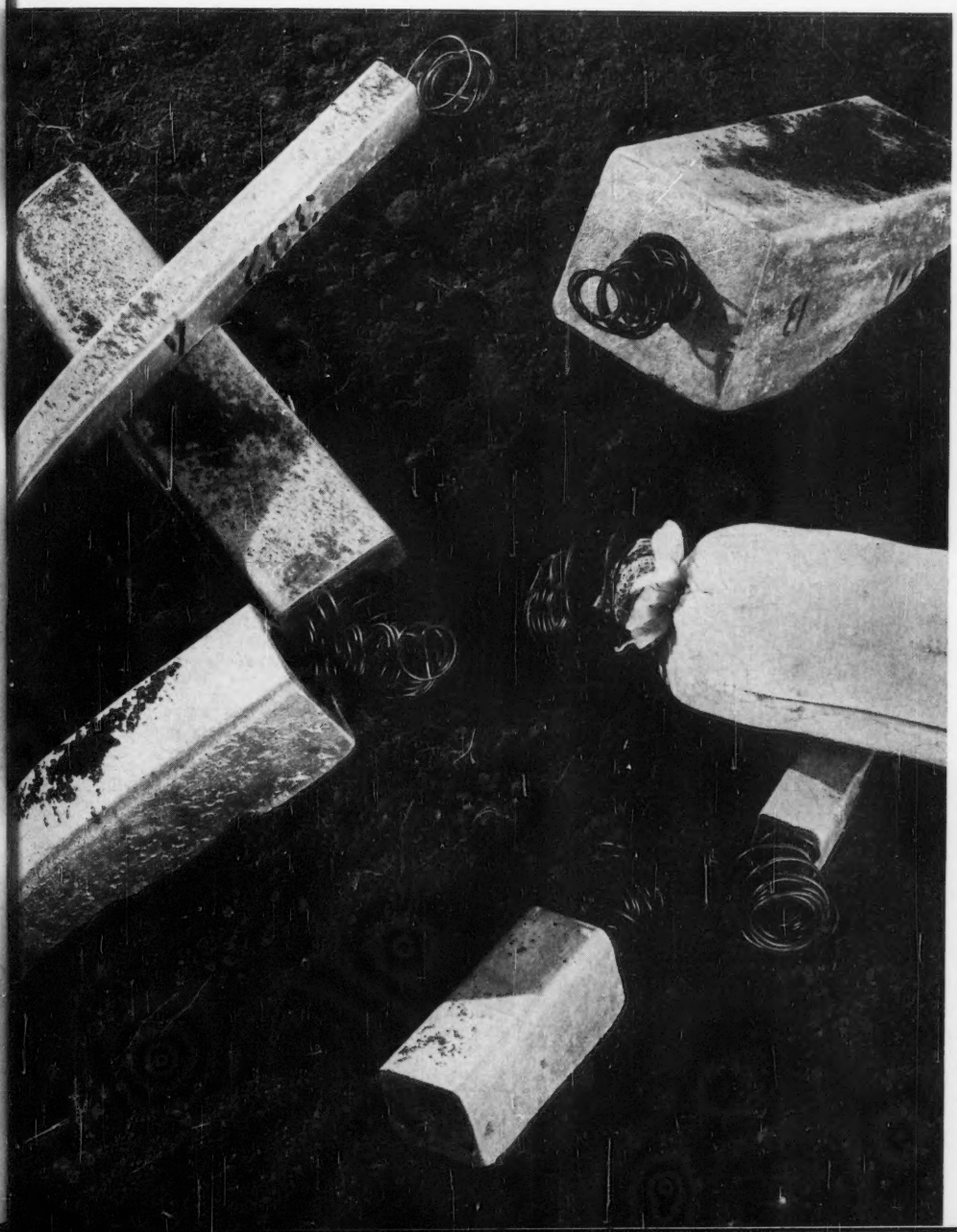

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AMERICAN SMELTING AND REFINING COMPANY

and H. P. Tripp. Ceramic Industry, **69,** No. 4, 82-84, (1957) October.

Enamels for 2S aluminum were developed which exhibited corrosion rates of about 1/75 that of bare aluminum when exposed to boiling alkaline water for 100 hours. These enamels were based on the sodium oxide-lead monoxide-silicon dioxide eutectic composition with substitutions of a total of 10 to 15 wt. per cent of titanium dioxide, zirconium dioxide, and alkaline earth oxides for lead monoxide and sodium oxide. About 1 wt. per cent nickel monoxide was beneficial for promoting adherence.—BTR. 15347

5.4.3, 3.5.4, 4.3.2

Asphalt Lining of Radiochemical Waste Storage Basins. Application of Asphaltic Membranes Appears Practical as Lining of Earth Storage Pits for Aqueous Radiochemical Waste. Clyde D. Watson, George A. West and Arnold J. Hoiberg. Ind. Eng. Chem., **50,** No. 8, 87A-91A (1958) August.

Membrane asphalt seems to offer the best lining protection for storage basins to be used in long-term retention of neutralized partially decayed radiochemical wastes, and asphaltic plank material is only slightly less desirable. Irradiation of these materials causes embrittlement and a maximum dose is discussed. Chemical attack of solutions containing nitric acid is very severe at temperatures of 150 to 225 F and use of membranes at these temperatures should be considered only for solutions which contain less than 1 percent strong oxidizing acids. Salt solutions or alkaline solutions can be confined at 200 F if the membrane softening point is 270 F; however, tests indicate poor resistance to alkalies.—NSA. 16671

5.4.5, 5.2.1, 8.9.5

Ships' Bottom Paints in Relation to Cathodic Protection. T. A. Banfield. Corrosion Technology, **5,** No. 8, 243-246 (1958) August.

The author first discusses briefly the cathodic protection of ships' hulls with zinc and magnesium anodes and by an impressed current, and then describes laboratory experiments to test the durability of paints on cathodically protected steel. Paints applied to the hull within 5 or 6 ft. of the point of attachment of an anode must be particularly resistant to alkalis and must have excellent adhesion. Good surface preparation, preferably by sand blasting or shotblasting, is essential and vinyl, bituminous and epoxy resin coatings have given good service. For areas farther away from the anodes, conventional ships' paints are said to be satisfactory. 3 figures.—ZDA. 16739

5.4.5

Effect of Pretreatment and Degree of Oxidation of Surfaces Before Painting on the Durability of Paints. K. F. Trägardh. Corrosion et Anticorrosion, **5,** 169-173 (1957); Chem. Abs., **51,** No. 18, 14285h (1957).

Presents the results of long-term corrosion tests performed in Sweden on steel samples bent so as to present surfaces at angles of 45, 90 and 180 to the outside atmosphere. It was observed that the duration of the protective power of paints depends directly on the surface preparation preceding painting. The more the surface is rusted, the deeper the treatment must eat into the surface. —RPI. 16374

5.4.5, 4.6.11

Red Lead in the Protection of Iron and Steel. Pt. 4. H. Masseille. Corrosion Prevention & Control, **4,** 37-39 (1957) March.

Durability of red lead linseed oil paints and their resistance to sea water; recommendations for surface preparation and application.—MR. 14734

5.4.5, 8.9.4

The Unique Features of Bituminous Emulsions and Their Possible Application on the Indian Railways. S. Ramanujam. Indian Rail. Tech. Bull., No. 124, 30-50 (1957); abstract: Rail. Res. & Eng. News, **5,** No. 89/90, 87 (1957).

Uses suggested include roof and floor coatings, surface dressing of railway platforms, protection of concrete and asbestos-cement, soil stabilization, treatment of sleepers, anti-corrosive treatment of bridges, rails and fastenings, protection of the inside of steel panels of all-steel coaches, binders for indigenous slag wool insulation, anti-sweat coatings, sound-deadening paints and underbody protective coating for trucks. The manufacture of bituminous emulsions is also discussed.—RPI. 14811

5.4.5

Blistering and Flaking of Paint Films. Pt. II. J. W. De Wit. Verfkroniek, **31,** No. 4, 154-159 (1958).

Further natural exposure tests of a wide range of paints on wooden surfaces are reported from the aspect of blistering and flaking tendencies. Comparisons are made between classical oil paints and alkyd paints.—RPI. 16728

5.4.5, 1.3

Marine Paints. J. C. Kingcome. Anti-Corrosion Manual, **1,** 196-203 (1958).

A general survey of marine paints, with 12 references. Proper surface preparation, including the removal of mill scale, is stressed. In underwater paint systems, in general, the protective performance is proportional to the thickness of paint applied. The use of wash primers on metal surfaces before the application of the rest of the anticorrosive system improves the protective performance of the systems. Red lead is not suitable as a primer for underwater surfaces but is excellent for atmospheric conditions. Zinc chrome primers may be employed for all conditions, exterior and interior. Paints based on vinyl resins appear to perform well under marine conditions.—RPI. 16566

5.4.5, 6.6.7

Neoprene and "Hypalon" Synthetic Rubber in Corrosion Resistant Coatings. Corrosion Prevention and Control, **5,** No. 6, 41-45 (1958) June.

Reviews properties and applications of coatings compounded from Neoprene and Hypalon synthetic rubber. Outstanding property of these coatings is adhesion to substrate. Neoprene coatings have been used successfully on: ductwork, pipelines, exhaust blowers, rectifier rolls in paper mill, cavitated blades of water pump, condenser tube ends and tube sheets, tumbling barrels, wooden tanks, tank cars for caustic soda, coking plant equipment, coal chutes, hot acid fume scrubber, as well as on many types of marine equipment. Heat, chemical and weather resistance of Hypalon is discussed.—INCO. 16431

5.4.5

Painting Metals with Aluminium-Pigmented Finishes. A. Painter. Trav. Peint., **12,** No. 7, 264-265 (1957).

The formulation of aluminum powder pigmented paints for iron, aluminum and zinc surfaces is discussed.—RPI. 16270

5.4.5

Corrosion-Resistant Coatings—Natural Chlorinated and Synthetic Rubber. E. G. Rawlings. Corrosion Technology, **4,** 283-286 (1957) August.

Coatings for protecting steel and other metals from corrosive environment.—MR. 15038

5.4.5

Red Lead in the Protection of Iron and Steel. Pt. VI. H. Masseille. Corrosion Prevention and Control, **4,** 41-43 (1957) Sept.

Health safeguards; preparation methods; specifications recommended.—BTR. 15388

5.4.5, 3.5.9

Regulating Paint Temperature in Continuous Coating Operations. R. K. Tyson. Paint Ind. Mag., **72,** No. 10, 70-71, 84 (1957).

Problems affecting the need for cooling of paint in dipping tanks due to heating up by parts passing from a drying oven into the dipping tank are discussed.—RPI. 16430

5.4.5

Sheet-Metal (Coated with) Plastic. H. Kalpers. Plastverarbeiter, **8,** No. 11, 406-409 (1957); Brit. Plastics Fed. Abs., **13,** No. 2, 132 (1958).

Methods are given for the production of polyvinyl chloride coated sheet metal which can be processed in the same way as the uncoated metal. Some processing methods are considered and a number of applications of the materials given.—RPI. 15926

5.4.5

Anticorrosive Protection of the Metallic and Concrete Surfaces of the Backman Apparatus. A. I. Reibman, M. I. Finkel'shtein and R. P. Joffe. Khim. Prom., No. 6, 373-375 (1957).

The metal parts of the Backman apparatus receive a good protection by application of paints based on divinyl acetylene polymers in 7-8 mm. film thickness, dried at 100 C. The concrete surfaces are covered with two layers of an enamel pigmented with ferric oxide, three layers of a lacquer mixed with asbestos in 3:1 ratio and two layers of the lacquer alone. The results of several tests are given.—RPI. 16302

5.4.5

Polyamide Protective Coatings. Don E. Floyd. Materials and Methods, **45,** 136-139 (1957) May.

Polyamide resin films can be used to protect metals or plastics from abrasion, heat, water, grease, acids, and alkalies. —BTR. 14626

5.4.5

Significance of Pigments in Coloring and Protection Against Corrosion. (In German.) J. D'Ans and H. J. Schuster. Farbe u. Lack, **64,** No. 2, 73-77 (1958) February.

Corrosion prevention with red lead oxide and zinc powder.—MR. 16492

5.4.8, 4.2.7

Experience of Czechoslovak Industry in Protecting Equipment to be Exported to Tropical Countries. (In Russian.) A. V. Ryabchenkov, V. M. Nikiforova, N. B. Nezvanova and B. D. Samuylenko. Metalloved. i Obrabotka Metallov, No. 6, 59-63 (1957) June.

Various methods and materials used as protective covering on merchandise and machinery against exposure. Some protective coatings are heavily charged with fungicide.—MR. 16319

5.4.8

Anti-Corrosive Paints. (In Italian.) Carlo Rossi. Vernici, **14,** 51-67 (1958) Jan.

Types of anti-rust paints, composition, vehicles, pigments, behavior in service. —MR. 16586

5.4.9, 3.8.4, 6.2.3

Kinetic Studies on Formation of Black-Oxide Coatings on Mild Steel in Alkaline Nitrite Solutions. Ray M. Hurd and Norman Hackerman. Defense Research Lab. and Dept. of Chemistry, The University of Texas. J. Electrochem. Soc., **104,** No. 8, 482-485 (1957).

The formation of black-oxide (magnetite) coatings on mild steel in hot concentrated solutions of sodium hydroxide and sodium nitrite was studied over the temperature range 130—150 C. The following mechanism is proposed for the over-all reaction:

$$1)\ Fe \xrightarrow[OH^-]{NO_2^-} Fe(OH)_2$$

$$2)\ Fe(OH)_2 \xrightarrow[OH^-]{NO_2^-} Fe(OH)_3 \rightleftarrows FeO_2^-$$

$$2a)\ Fe(OH)_2 \rightleftarrows Fe^{++} + 2OH^-$$

$$3)\ Fe^{++} + 2FeO_2^- \rightarrow Fe(FeO_2)_2$$

Of the total iron dissolved in step 1), only about 30 percent was converted to black oxide on the surface. Values of the rate constant for step 1) were obtained from plots of total iron dissolved vs. time. Plots of the initial rates (oxide-free surface) vs. 1/T were linear, with an energy term of 41 kcal/mole. As the oxide coating built up, the rates dropped to a fraction of the initial rate and the energy term increased sharply. For thicker films the rates are apparently controlled by diffusion of the reactants through the film. 15311

5.8 Inhibitors and Passivators

5.8.2

Rust Inhibitors. (In German.) H. Spindler: Fertigungstechnik, **7,** 429-430 (1957) Sept.

Determination of exact amount of the rust inhibitor required is main obstacle in application. Its usability must be determined in practice. 10 references.—MR. 16312

5.8.2, 4.4.7

Development of High Temperature Oxidation-Corrosion Inhibitors to Improve Stability of High Temperature Hydraulic Fluids and Lubricants. Quarterly Progress Report No. 1 for March, April and May, 1957. Robert Brunier and Edward J. Schwoegler. Illinois Inst. of Tech., Chicago. Armour Research Foundation Project C103. U. S. Atomic Energy Commission Pubn., Sept., 18, 1957, 18 pp. Contract AF33(616)-5101.

Data were collected on known corrosion inhibitors for synthetic diester fluids, mineral oils, silicone and silane fluids and analyzed for indications as to structural configurations and functional groups contributing to the inhibitory effect. A synthesis program for inhibitors for synthetic diester fluids and silicones is described.—NSA. 15721

5.8.3

A Catalytic Mechanism of Anodic Inhibition in Metallic Corrosion. R. A. U. Huddle and P. J. Anderson. Atomic Energy Research Establishment, Harwell, England. Advances in Catalysis, **9,** 393-397 (1957).

It is well known that quite low concentrations of certain oxygen-containing anions, such as chromate, are effective inhibitors of aqueous corrosion of a number of metals. These results have been ascribed to specific adsorption of the inhibitor at anodic sites of the metal surface, or alternatively to continuous repair of the protective film. Work on the corrosion of aluminum has suggested that its resistance is largely dependent on the structure of the film; thus, complete passivity is considered to be associated with the formation of an oxide, while a hydrated oxide or hydroxide film only confers partial protection. It is postulated that the important factor in inhibition by oxygen-containing anions is their ability to cause the true oxide to be produced rather than the hydroxide which would otherwise form. It is thought that the anion acts catalytically in donating its own O^{2-} ion to the film in competition with the hydroxyl ion from the solution. These ideas are shown to apply to other metals in addition to aluminum and explain a number of facts which are inconsistent with existing theories of anodic inhibition. (auth).—NSA. 15815

5.8.4

The Passivating Properties of Pigments. (In Russian.) I. L. Rozenfel'd, et al. Bull. Acad. Sci., USSR, Chem. Sci. Div. (Izvest Akad. Nauk SSSR, Otdel. Khim. Nauk), No. 6, 679-683 (1958).

Passivating action of chromate pigments on steel was studied electrochemically. 4 figures, 3 references.—ATS. 16584

5.8.4, 4.3.2, 6.2.2

Effect of Inhibitors and Wetting Agents on the Corrosion of Iron in Dilute Sulfuric Acid. (In German.) W. Machu and Solomon El Gendi. Metalloberflache, **11,** 135-139 (1957) April.

Use of aryl-alkyl derivatives of dodecylsulphonates in dilute sulfuric acid. Action of inhibitors can be positively or negatively influenced by wetting agents. 9 references.—MR. 14818

5.9 Surface Treatment

5.9.3

Theory and Practice of Flame Spraying. K. W. Sippell. Werkstoffe u. Korrosion, **8,** No. 4, 185-216 (1957).

A detailed discussion with 78 references.—RPI. 14774

5.9.3, 5.4.5

Protection of Structural Steelwork in Chemical Plant. Mark Waghorn. Corrosion Technology, **5,** No. 4, 103-105 (1958) April.

Surface preparation by blast cleaning, flame cleaning or power tool cleaning. Classification of exposure conditions; suitable organic protective coatings.—MR. 16708

5.9.3, 5.4.3

Surface Preparation for Protective Linings and Coatings Used in Critical Service. S. J. Oechsle, Jr. & K. G. Le Fevre. Eng. Dig., **18,** 485-486 (1957) Nov.; J. Iron and Steel Inst., **189,** Pt. 2, 190 (1958).

Design is briefly considered and sand blasting, etching and cleaning are reviewed.—RPI. 16262

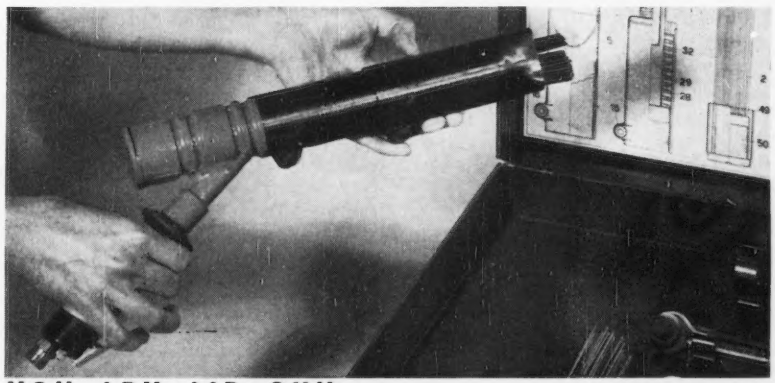
5.9.3, 3.5.3

Magnetostriction Vibrator Testing of the Effect of Shot Peening on the Cavitation-Corrosion Resistance of Metals. (In Russian.) L. A. Glikman and Yu. E. Zobachev. Metalloved. i Obrabotka Metall., No. 5, 38-41 (1957) May.

Investigation did not disclose positive influence of shot peening on resistance to vacitation corrosion. 5 references.—MR. 15788

5.9.4, 2.3.6, 6.4.2

Electron Micrographs from Thick Oxide Layers on Aluminium. C. J. L.



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Booker, J. L. Wood and A. Walsh. Brit. J. Applied Physics, **8,** No. 9, 347-352 (1957) Sept.

Electron micrographs have been obtained of the pore structure in the thick oxide layer formed by anodizing aluminum in sulfuric acid. The pores are shown to be minute tubes of approximately 200 Å. diameter running perpendicularly through the oxide and ending almost in contact with the underlying metal. The pore base thickness has been estimated, and the effect of a change in anodizing conditions on the pore base pattern is shown. (auth).—ALL. 14949

5.9.4

Nonmetallic Inorganic Protective Films. (In German.) G. Elssner. Metallwaren-Industrie und Galvanotechnik, **48,** 324-327 (1957) August.

Processing and purpose of phosphate chromate and oxide films on iron, zinc, aluminum and magnesium.—MR. 15774

5.9.4

Some Current Applications of Phosphate Coatings. P. Koeckritz, Jr. Plating, **45,** No. 3, 248-251 (1958).

Successful applications of phosphate coatings including their use as a paint base for cars, etc., are reviewed.—RPI 16726

6. MATERIALS OF CONSTRUCTION

6.2 Ferrous Metals and Alloys

6.2.2, 3.8.3

Studies on the Corrosion of 13% Chrome Steel. (In English.) Ryocho Wada. Hokkaido Univ., Faculty of Engineering, Memoirs, **10,** 471-490 (1957) Sept.

A major cause of the excellent corrosion resistance of an iron-chromium alloy containing more than 13 percent chromium is the rapid decrease of the critical current density required for its passivation. A logarithmic relation is found between the critical current density for passivation in 0.05 normal nitric acid and mole fraction of the alloy.—BTR. 16388

6.2.5, 3.5.9

Investigation of Iron-Manganese-Chromium-Nitrogen-Carbon System for Heat Resistance and Oxidation Resistance Between 1200 F. and 2000 F. Period covered January 1956 to December 1956. Chi-Mei Hsiao and Edward J. Dulis. Crucible Steel Co. of America. U. S. Wright Air Development Center, U. S. Air Force, Sept. 4, 1957, 157 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131563).

A comprehensive study of wrought chromium - manganese - carbon - nitrogen steels was conducted with the objectives, of developing outstanding elevated-temperature steels that contain a minimum amount of strategic alloying elements, and of advancing the existing knowledge of this new type of steel. Based on the results of the present investigation, phase boundaries of the base compositions of stable austenitic chromium-manganese-carbon-nitrogen steels have been established, and the effects of these elements and of vanadium, tungsten, molybdenum and niobium on the microstructures, and the room- and elevated-temperature properties of the steels have been evaluated. A nomograph was prepared to facilitate the designing of the base compositions of these steels, and the estimating of their room- and elevated-temperature properties. These steels are not as resistant to oxidation as are the chromium-nickel steels at comparable chromium levels. However, chromium - manganese - carbon - nitrogen base steels that have properly balanced compositions possess an excellent combination of strength and ductility at both room- and elevated-temperatures. For example, a steel containing 0.6 carbon, 12 manganese, 24 chromium, 0.7 nitrogen, 0.4 vanadium, 2 tungsten, and 0.9 niobium, had a 185,000 psi tensile strength, a 120,000 psi yield strength, a 48 percent elongation, and a 40 percent reduction of area. Also, its rupture strength was superior to those of 16 chromium-25 nickel-6 molybdenum, N155, 19-9DL, and A-286 in the temperature range of 1200 to 1400 F. (auth) —NSA. 15302

6.2.5, 8.4.5

Re-Evaluation of Chrome Plated Hardened Type 410 Stainless Steel for Slip-On Stator Type Mechanism Motor Tube. R. G. Christman, M. A. Golik, S. A. Greenberg, H. L. Helmbrecht, K. Jakobson, H. J. Kaplan, H. F. Mason, G. R. McIndoe, E. Rau, P. N. Seran, H. P. Wanter and M. O. Weaver. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-CTA(ME)-223, April, 1958, 54 pp. Available from Office of Technical Services, Washington, D. C.

The Submarine Thermal Reactor Mark I Core-3 utilizes hardened type 410 stainless steel as primary coolant pressure boundary material in the motor tubes of all control rod drive mechanisms. Stress corrosion data, other lab test data, and service performance data are reviewed, and the suitability of type-410 stainless steel for this application is re-evaluated.—NSA. 16464

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.6

Advantages of the Use of Cupro-Aluminiums in the Chemical and Allied Industries. Pierre Weill Couly. Génie chim., **77,** No. 4, 106-113 (1957).

Mechanical properties of aluminum bronzes (aluminum 4-12, manganese + iron + nickel $\geq$ 15 percent, balance copper) are reviewed, and the wide range of characteristics obtainable by small changes in composition is exemplified. Data on corrosion behavior, cavitation-erosion resistance, and weldability are presented, with special reference to applicability of the alloys to chemical-industry and similar requirements.—MA. 14930

6.3.6, 3.8.4, 3.4.7

An Investigation of Chemical Variables Affecting the Corrosion of Copper. W. D. Robertson, V. F. Nole, W. H. Davenport and F. P. Talboom, Jr. J. Electrochem. Soc., **105,** No. 10, 569-573 (1958).

In the presence of excess oxygen the corrosion rate of copper is a linear function of hydrogen ion concentration for each of three anions ($SO_4^=$, Cl^-, $C_2H_3O_2^-$). The rate is not a simple function of anion concentration, and the concentration dependence varies with pH. The effect of carbon dioxide is negligible. The temperature dependence of the rate, at constant oxygen concentration, for both chloride and sulfate solution, is

$$\text{Rate} = Ae^{-9000/RT}$$

The corrosion rate of copper in open solutions saturated with air goes through a maximum in the range 71-77 C. 16624

6.3.6, 7.3

The Development of High-Tensile Aluminum Bronze Alloys for Marine Propellers in Great Britain and the United States of America. F. Hudson. Intern. Shipbuilding Progress, **5,** 139-147 (1958) March.

Reasons for adoption of nickel-aluminum bronze alloys (e.g. Novoston) for marine propellers are high mechanical properties combined with resistance to corrosion and erosion. Notes on technical and production factors involved practical applications and results of preliminary sea trials are given.—INCO. 15671

6.3.10, 2.3.9

Direct Measurement of the Velocity of Propagation of a Ferromagnetic Domain Boundary in "Perminvar." E. W. Lee and D. R. Callaby. Nature, **182,** No. 4630, 254-255 (1958) July 26.

Using modification of Kerr magneto-optic effect, authors succeeded in measuring directly velocity of domain wall in magnetically annealed Perminvar (45 nickel-30 iron-25 cobalt). Observations showed that a large part of total change in magnetization took place by radial movement of singular circular wall from inside edge outwards. Graphs.—INCO. 17130

6.3.13, 6.3.15, 5.3.2, 4.3.7

Controlling Corrosion. T. J. Hull. Ind. & Eng. Chem., **49,** No. 1, 103A-104A (1957).

A short review of materials, including titanium and tantalum, of increasing use

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Ice Company, Chicago, Illinois Carbon dioxide condenser service.	.03 (18 years)	.05 (10 years)
Film Corporation, Binghamton, New York Tubes used in cooling service.	.04 (15 years)	.06 (8 years)
Industrial Plant, New Bedford, Massachusetts Ammonia condenser with salt water in coolant.	.04 (12-14 years)	.48 (1 year)
Industrial Plant, Toronto, Canada Tube and shell type condenser used for cooling brine with ammonia.	.01 (50 years)	.07 (7 years)
Ice Company, Aiken, South Carolina Ammonia condensers with mild brine solution containing 76 ppm sodium chloride for cooling.	.03 (18 years)	.29 (2 years)
Ice Company, Rocky Mount, North Carolina Condensers with mild brine solution containing 75 ppm sodium chloride for cooling.	.07 (8 years)	.16 (3 years)
Industrial Plant, Wisconsin Vaporizers for production of butane and propane gas. Severe stresses created by wide temperature variances.	.28 (2 years)	.96 (6 months)
Oil Company, Long Island City, New York Cool, weak ammonia liquor cools hot, concentrated ammonia liquor coming from generator on the way to absorber.	.03 (20 years)	.07 (6-8 years)
Process Company, Detroit, Michigan Heat exchangers with ammoniated brine concentration of about 10% ammonia and salt almost to saturation point.	.08 (8 years)	.16 (3 years)
Gas Company, Brooklyn, New York Ammonia condenser with cooling medium of creek water contaminated by the refuse from several nearby industrial plants.	.05 (16 years)	.22 (3-5 years)
Alkali Company, Saltville, Virginia Heat exchanger carrying ammonia liquor that is subsequently cooled by surrounding water.	.05 (16 years)	.30 (6 mos.-3 years)
Industrial Plant, Chicago, Illinois Condenser tubes in absorption system. Ammonia inside the tubing is about 200°F. and water outside the tubes is about 40°F.	.09 (9-11 years)	.28 (3 years)
Electric Utility Company, Hunts Point, New York Gas cooler drawing water from East River in New York City.	.15 (8-9 years)	.45 (2-3 years)
Warehouse & Cold Storage Corp., Springfield, Massachusetts Ammonia condensers and direct expansion cooling coil.	.02 (30 years)	.04 (12 years)

"Cost per foot per year" figures are calculated from current prices of cold drawn seamless steel tubing (ASTM A-179) and cold drawn wrought iron tubing (ASTM A-382). Because of the many variables involved, costs for installation labor, maintenance and replacement are not included. If they were, wrought iron's low cost story would, of course, be even more impressive.

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in corrosive environments. Aluminum coatings prevent corrosion by hydrogen sulfide.—MA. 15312

6.3.15, 3.7.2, 5.3.4

Status of an Alternate Control Materials Program. R. W. Dayton. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-X-135 (Del.), Jan. 15, 1957 (Declassified with Deletions March 7, 1957), 7 pp. Available from Office of Technical Services, Washington, D. C.

A study being made of the tensile properties and corrosion resistance of several boron and B_4C dispersions in titanium is summarized. A study of dispersions of ZrB_2 in titanium to determine the optimum loading and particle size of the ZrB_2 necessary to obtain the best corrosion resistance mechanical properties and core-to-clad bond is currently in progress. Development work on fabrication of full-size control-rod blades is reported. Cladding of boron-titanium dispersions with Zircaloy-2 is discussed as possible alternate control rod material.—NSA. 16156

6.3.15, 3.5.9, 1.6

The Properties of Titanium Alloys at Elevated Temperatures. F. R. Schwartzberg, F. C. Holden, H. R. Ogden and R. I. Jaffee. Titanium Metallurgical Laboratory. Battelle Memorial Institute, Office of Assistant Secretary of Defense for Research and Development. September, 1957, 228 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 121634).

This volume contains an accumulation of data dealing with the mechanical properties of titanium alloys at elevated temperatures. The information was collected from the literature on the subject. The properties presented include short-time strengths and creep and rupture strengths, as well as less complete data on bearing, shear, and notch strengths. A discussion of thermal stability also is included.—OTS. 15054

6.3.15, 3.5.9, 8.9.1

A Brief Study of the Suitability of Titanium and a Titanium Alloy as Firewall Material. Civil Aeronautics Administration Technical Development Rept. No. 317. C. A. Hughes. U. S. Wright Air Development Center, U. S. Air Force, Sept., 1957, 10 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131392).

Use of commercially pure titanium and titanium alloy in the construction of aircraft powerplant firewalls, ducts, and shrouds has resulted in a need for information on the structural strength and behavior of these materials when subjected to high temperatures and flames. Tests were conducted on several grades of titanium and one alloy to determine strength at high temperatures and resistance to a 2000 F. flame. Comparison tests of stainless steel were also made. Among the results, it was indicated that the strength of titanium and titanium alloy decreased as temperature of the metal increased. Stainless steel remained stronger throughout the 1000 to 1800 F. range of test temperatures. However, the rate of decrease in strength progressed more rapidly in stainless steel than in titanium, until at 1800 F. the difference in strength was not as pronounced as at 1000 F. Titanium panels as thin as 0.016-inch resisted a 2000 F. flame for a minimum of 15 minutes without visible deterioration.—OTS. 15305

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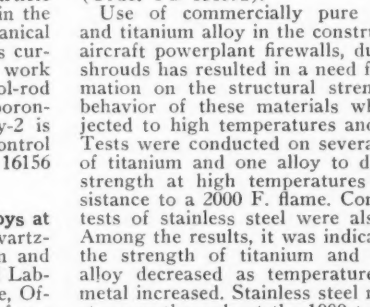

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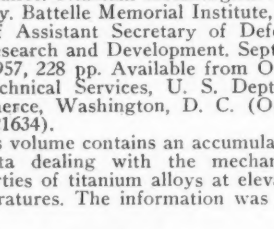

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6.3.15

Platinum-Faced Titanium for Electrochemical Anodes. J. B. Cotton. Platinum Metals Review, **2**, No. 2, 45-47 (1958) April.

Titanium is resistant to the passage of current into an electrolyte, but has low resistivity when a second metal makes contact with it. When titanium is used as an anode in salt water and an anodic current increased, the voltage required to force the current through the service film rises until, at the weakest point, the film breaks down and local corrosion ensues. If, however, a small piece of platinum foil or wire is spot welded to the titanium most of the current passes out through the platinum and the titanium is uncorroded.—MR. 17195

6.3.17, 4.6.1

Development and Properties of Uranium-Base Alloys Corrosion Resistant in High Temperature Water. Part I. Alloys Without Protective Cladding. W. A. Bostrom, M. W. Burkart, E. K. Halteman, R. D. Leggett, R. K. McGeary and T. R. Padden. Westinghouse Electric Corporation. U. S. Atomic Energy Commission Pubn., WAPD-127, April 25, 1955 (Declassified Feb. 6, 1957), 164 pp. Available from Office of Technical Services, Washington, D. C.

The status of work is presented on the development of uranium-base alloys that are corrosion resistant in high temperature water. Binary and ternary alloys of molybdenum, niobium, silicon, zirconium, germanium and other elements have been studied and their properties, phase relationships and corrosion resistance are summarized.—NSA. 15734

6.3.19, 5.3.2, 8.1.2

Corrosion of Metals in Buildings: The Performance of Zinc and Zinc Coatings. R. W. Bailey and H. G. Ridge. Paper before Soc. Chemical Industry Symposium on "Corrosion of Metals in Buildings," London, March 21, 1957. Chemistry and Industry, No. 37, 1222-1227 (1957) Sept. 14.

Discusses performance of zinc and zinc sheet products in and on buildings, with steel protected from rust by means of a zinc coating, and behavior of zinc alloy die-cast builders' hardware. Graph, table.—INCO. 14948

6.3.19, 3.2.3

Zinc Surfaces After Various Preparative Treatments. (In German.) K. Huber and D. Laske. Metalloberflache, **11**, 217-225 (1957) July.

Surface layers resulting from air, water, or aqueous solutions are composed almost entirely of zinc oxide, show very different but reproducible structures depending on the corroding material.—BTR. 14696

6.3.20, 4.6.2, 4.7

Corrosion Properties of Zirconium and Zirconium Alloys. M. H. Boyer. California Research and Development Co. U. S. Atomic Energy Commission Pubn., CRD-T2C-63, October 23, 1951 (Declassified April 2, 1957), 8 pp. Available from Office of Technical Services, Washington, D. C.

Zirconium is resistant to corrosion in sodium-potassium up to 500 C, but it shows high resistance to water corrosion up to 600 F and to superheated steam up to 750 F if it is free of impurities. Zirconium alloys with 3 to 6 percent tin show a high resistance to water up to

600 F and to superheated steam to 750 F and are not seriously affected by the minor amounts of impurities which affect pure zirconium. Welding and bonding zirconium and zirconium-tin alloys produce corrosion resistant joints if the surfaces are properly prepared and atmospheric gases are excluded during the processes.—NSA. 15719

6.3.20, 3.4.9

Evaluation of Tests for Classification of Zirconium for Corrosion Resistance. D. M. Wroughton, P. Cohen and D. J. DePaul. Westinghouse Electric Corp. U.S. Atomic Energy Commission Pubn., WAPD-RM-37, Jan. 19, 1951 (Declassified March 11, 1957), 9 pp. Available from Office of Technical Services, Washington, D. C.

Recent studies show that 20 to 30 percent of the zirconium which passes the two week water classification test at 600 F fails in 50 to 60 days on continued testing. About 5 percent shows serious failure. Work has been started to study the feasibility of testing zirconium in water at 680 F with the hope that the metal surviving a short-time test at this temperature would withstand 600 F water for an extended period of time. (auth)—NSA. 16348

6.4 Non-Ferrous Metals and Alloys—Light

6.4.1

Light Metals and Alloys in Corrosive Media. (In French.) J. Herenguel and Pierre Lelong. Nature, No. 3271, 445-450 (1957) Nov.

Nature of corrosion; laboratory methods of determining corrosivity; types of protection available; corrosivity of aluminum and magnesium alloys, beryllium, titanium and zirconium.—MR. 16176

6.4.2

Corrosion of Structural Aluminum and Measures for Its Prevention. (In Serbian.) Fran Podbreznik. Zastita Materijala, **5,** No. 11, 389-394 (1957) November. 15398

6.4.2, 3.7.3, 4.2.1, 4.6.11

Weldable Aluminum Magnesium Alloys. (In German.) P. Brenner. Schweissen und Schneiden, **9,** 483-492 (1957) Nov.

Corrosion resistance of welded and unwelded 4 percent magnesium and 5 percent magnesium sheet to atmosphere and sea water. Stress-corrosion sensitivity. 12 references.—MR. 15530

6.4.2, 7.5.1

Aluminium Aerosol Containers in Europe. Aksel Taranger. Light Metals, **20,** No. 236, 359-362 (1957).

A general survey of the development of impact-extruded rigid containers for aerosols. The metal is immune to attack by the contents, particularly when lacquered or anodized and will stand up to any pressure normally used.—MA. 16414

6.4.2

Corrosion Characteristics of Aluminum and Its Alloys. Pt. 2. R. F. Darnell. Chem. Ind. Eng., **9,** 19-23 (1957) August.

Effect of purity and alloying conditions on corrosion resistance; serviceability of aluminum under exposure to atmospheric attack, natural waters, sea waters, soil contamination, building materials, chemicals. 25 references.—MR. 16148

8. INDUSTRIES

8.4 GROUP 4

8.4.3, 3.4.8, 3.2.3

How Humble Runs Catalytic Reformers. H. C. Nix. Paper before API, Refining Div., Philadelphia, May 13-16, 1957. Petroleum Refiner, **36,** No. 5, 189-192 (1957) May.

Principal difficulties encountered in operation of platinum reformers were caused by sulfur and hydrogen sulfide corrosion of hydrocarbon piping, resulting in metal loss and scaling. Sulfur corrosion rates of carbon-½ molybdenum tubes of naphtha preheat plant were 200 mils per year; this has been retubed with 9 chromium-1 molybdenum. Hydrogen sulfide corrosion rates of 60-90 mils per year were experienced in low-chromium-molybdenum reheat tubes and transfer lines. No corrosion was found in 18-8 tubes in converted reformer. Double block valves permit screening of catalyst to prevent plugging due to scale flaking. Permanent solution to corrosion and scale will be installation of hydride-sulfurization unit to reduce sulfur levels to less than 25 ppm.—INCO. 16263

8.4.3, 4.3.2, 3.4.8

Where and Why Alloys were Used. J. H. Proctor and A. Hoersch. Lukens Steel Co. Petroleum Refiner, **36,** No. 6, 150-152 (1957) June.

Alloy and alloy-clad steels were used for the backbone of Tidewaters' new Delaware refinery. Alloys used were types A204 (a carbon-molybdenum

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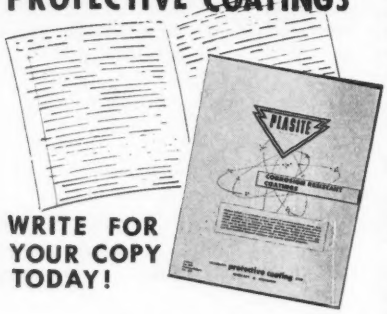

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alloy) and A301 (a chromium-molybdenum alloy). Clad steels were types 304, 304L, 316, 316L, 405, 410, Monel and a 78 nickel, 15 chromium, 6 iron alloy. Reactor scrubber used 316L stainless clad steels to resist corrosion from sulfur compounds and napthenic acids. Fractionator tower used Monel clad in upper section and 12 chromium stainless clad in lower section to resist corrosion from mineral acids and sulfur attack. Gasoline column used Monel clad steels in upper portion to resist corrosion from hydrochloric acid and other strong mineral acids. Gasoline stabilizer was constructed of steel clad with an alloy of 78 nickel, 15 chromium and 6 iron in lower section and Monel clad in upper section. Photos.—INCO. 16273

8.4.3, 3.4.8, 3.4.3

Corrosion of Steel Pipes in Refinery Equipment by Sulphur-Rich Crude Oil. (In German.) P. Csokan. Werkstoffe u. Korrosion, **9,** 443-450 (1958) July.

Corrosion and its thermal decomposition products. Chemical analysis and X-ray investigation of corrosion products. Corrosion mechanisms. 22 references.—MR. 16792

8.4.3, 3.7.3, 6.2.5

Selection, Welding of Chromium-Molybdenum Alloy Steel Pipe. J. Bland. Standard Oil Co., Indiana. Welding Engr., **43,** Nos. 7, 8; 34-36; 42-44, 46 (1958) July, August.

Discusses several determining factors in selection of chromium-molybdenum steel for petroleum refinery piping, namely—resistance to graphitization, to high temperature hydrogen attack, to oxidation or scaling and to corrosive attack, as well as requisite of higher strength at elevated temperatures. Effects of preheat and postheat treatment on weld properties of these steels are evaluated. Numerous graphs, photomicrographs, isothermal transformation diagrams.—INCO. 16778

8.4.5, 6.2.5, 3.5.4

Decontamination of Stainless Steel. D. O. Campbell. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-1826 (Del.), March 2, 1955 (Declassified with Deletions Feb. 22, 1957). 45 pp. Available from Office of Technical Services, Washington, D. C.

The contamination on stainless steel exposed to Purex process solutions is predominantly niobium, which is also the most difficult contaminant to remove. Zirconium accounts for a few percent of the contamination and other fission products are relatively unimportant. Contamination of stainless steel with various dilutions of Purex waste followed the Freundlich adsorption isotherm relation between the amount of radioactivity adsorbed and its concentration in the contaminating solution. The addition of a fluoride solution of stable niobium and zirconium to the contaminating solution decreased the gamma activity adsorption by decreasing that of niobium and zirconium. Ruthenium then became relatively important. The most effective noncorrosive decontaminant was alkaline tartrate-peroxide. After the initial application of this reagent, its action was improved by a pretreatment of the contaminated surface with nitric acid. Several alternate treatments with nitric acid and alkaline tartrate-peroxide gave the best decontamination with negligible corrosion. The most effective decontaminant, 3 per cent hydrofluoric acid in 20 per cent nitric acid, was severely corrosive to stainless steel. The rate of decontamination is reported for sodium hydroxide, alkaline tartrate, alkaline tartrate-peroxide, nitric acid and oxalic acid. The reagents decontaminated rapidly at first, but the rate dropped to about 6 per cent removal of activity per day after several days. High-intensity ultrasonic radiation increased the decontamination rate, but the problem of getting the radiation into large equipment would be formidable. A survey of the literature on decontamination is given. (auth)—NSA. 16197

8.4.5, 3.7.2, 6.2.5

Boron Steel for Control Rods and Thermal Shields. N. Balai. Argonne Nat'l. Lab. Nucleonics, **16,** No. 1, 100-101 (1958) Jan.

Of the 9 control rods in the EBWR, 4 have boron steel absorber sections (other 5 have hafnium). Thermal shield surrounding core is also of boron steel. Advantage of using boron steel for thermal shields derives from its ability to absorb neutrons with consequent reduction in formation of energetic capture gammas. Because of this, diameter of pressure vessel can be reduced by reducing radical thickness required for thermal shield. Development of specimens of alloys of natural boron and Type 304 and CF-8 stainless is described. Weldability and irradiation resistance of steels are discussed. Corrosion resistance of 1 and 2 wt. per cent boron stainless steel alloys is equal to that of Type 304. Photo.—INCO. 15180

8.4.5, 6.2.5, 4.7, 6.3.20, 4.6.2

Corrosion in Atomic Energy Installations. (In French.) Leo F. Epstein, H. Mogard, J. R. Weeks, C. J. Klamut, M. Silberberg, W. E. Miller, D. H. Gurinsky, etc. Metaux-Corrosion-Industries, **32,** 490-498 (1957) Dec.

Corrosion of stainless steel by liquid metals (sodium, bismuth-uranium, lead, lead-bismuth eutectics), and zirconium and alloys by water at high temperatures.—MR. 15772

8.4.5, 4.3.3

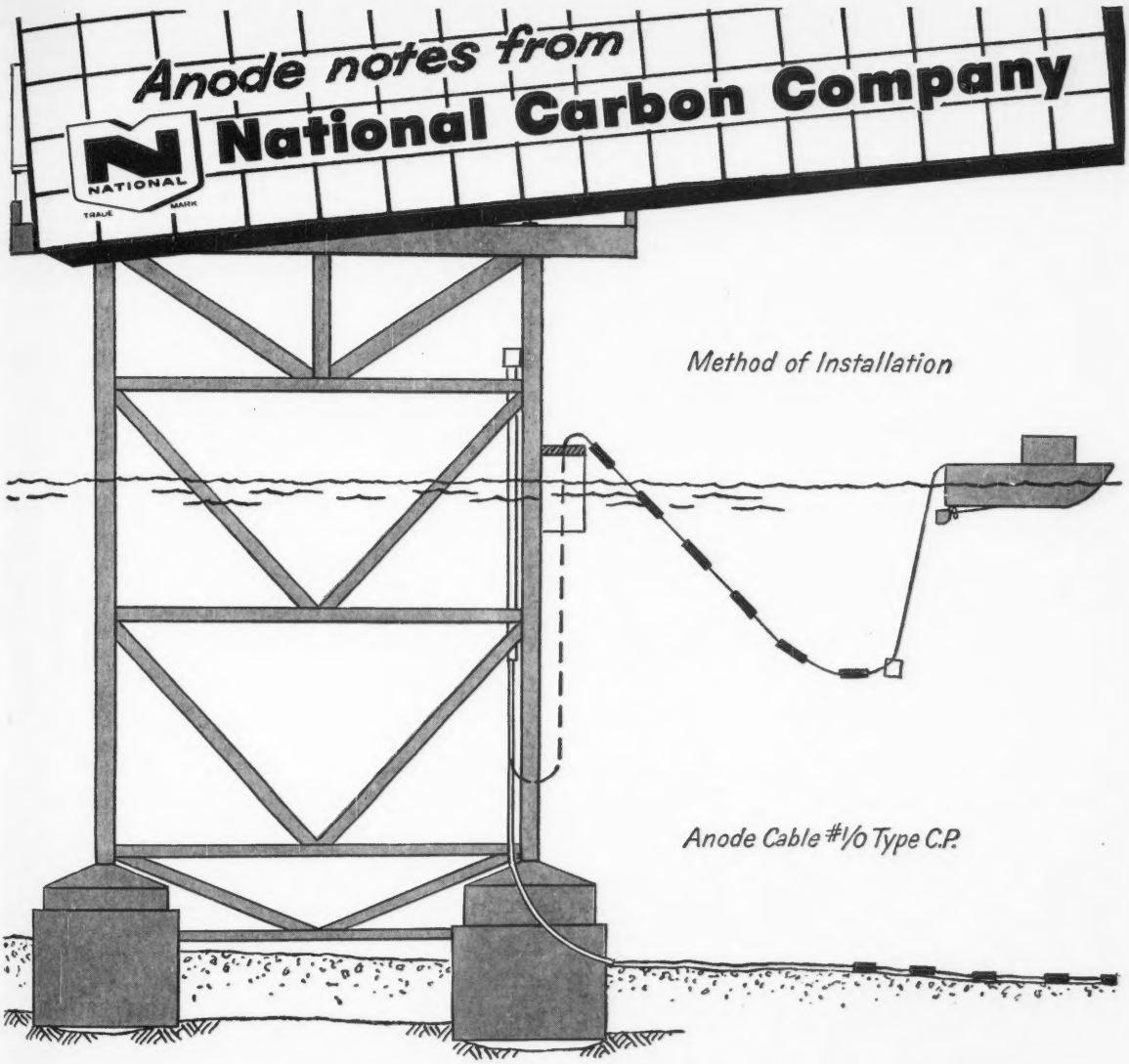
Report of Slurry Blanket Test Run SM-2. L. F. Parsly, Jr., H. L. Falkenberry and I. M. Miller. Oak Ridge National Lab. U. S. Atomic Energy Comm. Pubn., CF-57-4-87, April 29, 1957, 73 pp. Available from Office of Technical Services, Washington, D. C.

Run SM-2 was run to determine whether a sulfated thorium dioxide slurry could be handled in a hydrodynamic system similar to the HRT test blanket. It was found that the thorium dioxide concentration circulating in the pipes was consistently 50 per cent or less of the expected concentration based on the quantity of oxide changed. The run lasted 1730 hours. It was terminated because of a slurry leak a few days before a shut-down had been scheduled. Severe erosion was found in the pump impeller and flow nozzle. Chloride concentrations high enough to cause concern over possible stress corrosion cracking occurred on several occasions in the pressurizer.—NSA. 14741

8.4.5

Chemical Problems in Coolant Gases. W. E. Dennis. G.E.C./Simon-Carves Atomic Energy Group. Nuclear Eng., **2,** 321-323 (1957) August.

Processes involving the coolant gas such as the mass transfer of carbon, the oxidation of metals and the removal of



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moisture should not be viewed as isolated and independent phenomena. In the absence of further information on the effects of irradiation on chemical reactions occurring in the gas circuit only a qualitative picture can be obtained of the gas composition likely to be established during operation. Under what appear to be likely conditions in a carbon dioxide cooled graphite moderated reactor it may be shown that significant concentrations of hydrogen might be produced by the reduction of water with the carbon monoxide. The water-gas reaction will prevent the accumulation of hydrogen from extraneous sources but may render the removal of moisture more difficult. In the presence of very small concentrations of water vapor the formation of zirconium hydride is thermodynamically possible at all temperatures and cannot, therefore, be neglected in specifying conditions for oxidation tests. (auth)—NSA. 15268

8.4.5

Evaluation of Loop Components and Admixed Thorium-3% Uranium Oxide Slurry in 200A Loop (Summary of Run 200A-10). R. B. Gallaher, A. S. Kitzes, and C. F. VandenBulck. Oak Ridge National Lab. U. S. Atomic Energy Comm. Pubn., CF-57-6-67, June 13, 1957, 27 pp. Available from Office of Technical Services, Washington, D. C.

A slurry addition system, a venturi in the circulation loop, and two types of sampling systems were tested with 500 and 800 g thorium/kg water slurries in the 200 gpm loop at 250 C and 1000 psig. The addition system worked satisfactorily while the venturi gave erratic readings during part of the run. Both the capillary and in-line sampling systems proved satisfactory with the capillary sampler being much easier and more convenient to operate. The addition of uranium to the slurry had no appreciable effect on the handling characteristics, the attack rate on 347 SS, the particle size and crystallite size of the thorium dioxide. The attack rate was found to be 1 mpy during the first 100 hours of circulation and decreased to 0.4 mpy at the end of the run. (auth)—NSA. 14655

8.4.5

Effects of Sodium and Water Leaks on Experimental SDR Barriers. C. Bolta and J. O'Neill. Nuclear Development Corp. of America. U. S. Atomic Energy Commission Pubn., NDA-84-9, August 15, 1958, 45 pp. Available from Office of Technical Services, Washington 25, D. C.

Tests are described which are part of the experimental program on means of maintaining separation of sodium coolant and heavy water moderator in the SDR. A test section was built to simulate a portion of a fuel coolant tube with its encasing barrier tube assembly and calandria tube. Leaks of sodium and moderator from their containment were simulated by means of sodium and water jets directed against the barrier tube assembly. Double and single-walled barrier assemblies were tested. (auth)—NSA. 16752

8.4.5, 4.3.3, 6.3.20, 2.3.8, 5.11, 1.2.2

Homogenous Reactor Preliminary Process Design Report. Quarterly Report. Technical Division. C. B. Graham, C. H. Secoy, I. Spiewak, E. H. Taylor, A. M. Weinberg, C. E. Winters and J. L. English. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-527 (Del.), Dec. 28, 1949 (Declassified March 18, 1957), 110 pp. Available from Office of Technical Services, Washington, D. C.

A description and discussion of the general feasibility of a 200 to 1000 kw pilot model homogeneous reactor are presented. Topics discussed include design data, reactor control, cost estimates, and the corrosive effects of uranyl sulfate for zirconium. Schematic sketches illustrate the design proposals.—NSA. 15844

8.4.5, 6.2.5, 4.3.3

Value Test Loop Operation—November 23, 1955 Thru October 18, 1957. A. M. Billings. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-57-12-57, December 12, 1957, 12 pp. Available from Office of Technical Services, Washington, D. C.

The operation of the valve test loop for the evaluation of valve trim materials in uranyl sulfate solution is presented. Armco 17-4 PH stainless steel hardened from Rc 35 to Rc 40 appears best for a valve plug material when used with a 347 stainless steel valve seat. Other materials showing near-equal performance are the 347 stainless steel plug and seat with gold-gasketed tongue and groove and 347 stainless steel plug flame plated with Lende LW-1 working against an unplated 347 stainless steel seat.—NSA. 15548

8.4.5, 6.2.5, 6.3.15

Rare Earth Alloys for Control of Atomic Reactors. U. S. Bureau of Mines, Northwest Electrodevelopment Lab. U. S. Atomic Energy Commission Pubn., USBM-C-172, 3rd Quarterly Progress Rept. for Fiscal Year 1957 (Declassified March 4, 1958), 14 pp. Available from Office of Technical Services, Washington 25, D. C.

The work was concerned primarily with stainless steel and titanium alloys containing high percentages of gadolinium and europium. Data are given on corrosion, fabrication and melting points of these alloys. Photomicrographs showing alloy structure are included.—NSA. 16756

8.4.5, 3.5.8, 3.5.9, 6.2.5

What Can be Done to Combat Failures in Nuclear Piping? V. T. Malcolm and S. Low. Paper before 3rd Nuclear Eng. and Science Conference, Chicago, Mar. 17-21, 1958. Heating, Piping and Air Conditioning, **30,** No. 6, 112-114 (1958) June.

Discussion of causes and cures for thermal failures in nuclear piping. The classic test for proving both the pressure tightness and strength of piping system components is still the hydrostatic test of ambient room temperature. Most such tests are performed on hot or cold clean loops. In order to maintain loop water purity the loops are usually constructed of austenitic stainless steel with purification devices incorporated into the loop. Austenitic chromium-nickel stainless steel is generally used for nuclear power piping and components. This material has the unusual property of incorporating a high thermal coefficient of expansion with a low thermal conductivity. For the same heat transferred much higher thermal stresses can result than for carbon or low alloy steels. Most of the chromium-nickel austenitic stainless steels used for nuclear power piping originally are not notch sensitive and when an excessive load is applied, lateral contraction results with an apparent reduction of stress. Where the sum of the pressure and thermal stresses cannot be kept within the yield strength of the metal, ductility must be depended upon to relieve the peak stresses. During fabrication a condition exists in most welds of austenitic stainless steels where weld shrinkage produces residual stress considerably in excess of the yield strength but is relieved by plastic flow. Photo.—INCO. 16721

8.4.5, 4.3.2, 4.3.3

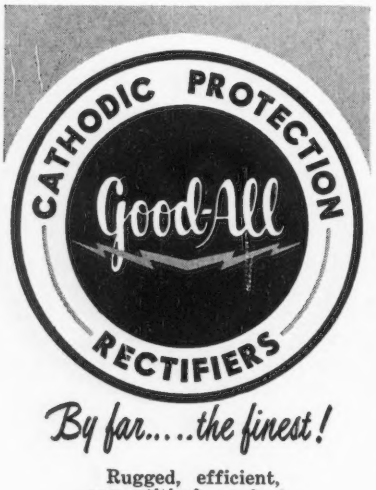
Problems Associated with the Chemical Processing of Aluminum-Silicon-Plutonium Fuel Materials. E. E. Voiland. General Electric Co., Hanford, Atomic Products Operation. U. S. Atomic Energy Commission Pubn. HW-54819, April 1, 1958, 13 pp. Available from Office of Technical Services, Washington, D. C.

The aluminum-plutonium-silicon system having been proposed as fuel material for the Plutonium Recycle Test Reactor, its behavior in chemical processes was investigated. The silicon reduced the dissolution rate and would not dissolve in nitric acid. A hydrofluoric acid-nitric acid system would dissolve it but equipment corrosion, volatile fluorides and other problems result. Dissolution in boiling sodium hydroxide results in a colloidal solution which resists coagulation. These and other findings lead to a recommendation not to use silicon.—NSA. 16684

8.8 Group 8

8.8.1

Design and Operation of Chlorine Dioxide Generating Plant at Penobscot. E. D. Thomas. Rust Eng. Co. Paper Trade J., **142,** No. 20, 30-32 (1958) May 19.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Description of Penobscot Chemical Fibre Co.'s recently completed chlorine dioxide generating plant. Chlorine dioxide gas is produced by mixing sodium chlorate, methanol and sulfuric acid to accomplish an oxidation-reduction reaction where chlorine in the sodium chlorate is converted to chlorine dioxide gas. Sodium chlorate is shipped in tank cars in an anhydrous crystalline form and dissolved with a measured quantity of warm water to form a 46 weight per cent solution stored in type 304 stainless steel tanks. Due to corrosive nature of chemicals involved, special piping materials had to be used as well as chemical resistant materials for the operating equipment. Saran-lined steel pipe was specified for chlorine dioxide bleach liquor, spent generator liquor and dilute sulfuric acid service. Polyvinyl chloride pipe was specified for chemical feed lines and generator insert nozzles. Diagrams, photos.—INCO. 16639

8.8.1, 8.4.5

The Excer Process for the Conversion of Uranyl Nitrate to Uranium Tetrafluoride. F. R. Bruce. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., CF-53-11-156, Nov. 25, 1953 (Declassified Feb. 21, 1957), 6 pp. Available from Office of Technical Services, Washington, D. C.

The Excer Process for the preparation of uranium tetrafluoride from aqueous uranyl nitrate is reviewed. Uranyl nitrate is converted to uranyl fluoride by ion exchange; then, uranyl fluoride is electrolytically reduced to hydrated uranium tetrafluoride. Advantages of the Excer Process include adequate fission-product decontamination, production of uranium tetrafluoride at a lower cost, and better separation of uranium from ionic impurities; the main disadvantages results from the handling of aqueous hydrofluoric acid and its associated corrosion problems. Cost estimates are included.—NSA. 15229

8.8.3, 6.3.10

The Anodic Behavior of Various Grades of Nickel in Plating Solutions. Pts. I and II. (In German.) E. Raub and A. Disam. Metalloberfläche, **12**, 161-167, 193-197 (1958) June, July.

Investigation into the anodic behavior of different grades of nickel in common nickel plating solutions: work on five types of nickel anodes (four rolled anodes and one electrolytic nickel anode including one depolarized, rolled anode: composition and micros of the anodes). Four types of plating solutions used for test were: nickel sulfate, nickel chloride, nickel sulfate-nickel-chloride and commercial bright nickel plating solution. Anode potential-current density curves were obtained under various conditions. Sludge formation is discussed.—INCO. 16621

8.8.5

Sintered Alumina Molds for Investment Casting of Steels. F. C. Quigley

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and B. Bovarnick. Trans. Am. Foundrymen's Soc., **66,** 247-251 (1958); Modern Castings, **33,** No. 6, 51-55 (1958) June.

Investigation was undertaken for development of new molding process for investment casting of steel alloys. Need for high refractoriness and low cost, coupled with processing requirements, led to selection of aluminum oxide as optimum mold material. Stable slurry was developed from aluminum oxide combined with binders and wetting agent. Mold is formed by alternately dipping wax pattern into slurry and sprinkling dip coat with coarse grain aluminum oxide until built-up wall of desired thickness is attained. Green mold is dried overnight in preparation for wax melt-out and firing. Melt-out is accomplished in less than two minutes and mold is fired at 2200 F for 25 minutes. After firing, molds have sufficient strength at 1200-1600 F to withstand mechanical and thermal shock of pouring. High-quality castings of several alloy steels, including 4340, have been produced with good surface, metal quality and dimensional control.—INCO. 16591

8.8.5

Study on Manufacturing a Large Composite Wheel, the Rim of Which is Made of Super-High Grade Heat-Resisting Steel LCN-155. Y. Ishihara, S. Yamaki and K. Deguchi. Paper before Iron & Steel Inst. Japan, October, 1956. J Iron Steel Inst. Japan, **43,** No. 2, 140-153 (1957) February.

LCN-155 was melted in a 2-ton high frequency induction furnace and cast into octagonal 350-kg. ingot. The ingot after diffusion annealing, was forged and swaged with utmost caution. Cracks occurred, however, at the edge of the ingot during swaging by 2000-ton hydraulic press. Cracks occurred at the "inner wrinkles" caused by segregation of impurities such as carbides. Alloy was remelted in a 2-ton induction furnace. This ingot, after reheating, was swaged, core-punched and ring forged. Cracks on the rim were prevented. Rim was solution treated at 1200 C. and the boss was annealed before welding. After welding of the composite parts, it was heat-treated at 800 C x 24 h. followed by furnace cooling for the sake of stress-relieving and aging. For welding of composite parts, AISI 309 niobium welding rods were first used. Since they were not good enough to make a perfect weld, they were replaced by AISI 310 rods. The weld was not satisfactory. Rim and boss were again prepared together with LCN-155 welding rods (5 mm. in diameter) and welded by argon-arc tungsten method, giving satisfactory results. High temperature mechanical testing confirmed that the strength of the rim which was exposed to high temperatures and the strength of the boss which was kept at low temperatures were well balanced.—INCO. 15329

8.8.5

Designing Vacuum Plaster-Mold Castings. W. G. Wilkins. Universal Castings Corp. Machine Design, **29,** No. 24, 106-108 (1957) Nov. 28.

Vacuum, applied to plaster-molds, produces castings with unusual physical properties without sacrifice of intricate detail or close dimensional tolerances. To avoid oxidation of alloys, pouring is done at reduced temperatures. Rapid solidification results, producing a close-grained, dense casting with porosity and inclusions virtually eliminated. At low temperatures, metal is viscous and sluggish. Therefore, to achieve intricate details and close configurations, a partial vacuum is applied around both cope and drag sections of mold. Materials vacuum cast include brass, aluminum bronze, nickel brass and high-strength aluminum. One of the alloys most frequently used in food-processing industries is a 22 nickel brass, commonly known as "dairy metal." Due to smooth surface produced by plaster-mold vacuum casting, little polishing is required. When polished, finish of this metal blends well with stainless steel. Dense cast surface, when highly polished, eliminates porosity where bacteria might lodge. Corrosion resistance is excellent. Photos.—INCO. 15141

8.8.5, 3.7.3, 3.7.4

Investigations Concerning Grain Boundary Brittleness in Intricate Die-Castings Made of Aluminium-Zinc-Magsium-Copper Alloy Materials. (In German.) Wilhelm Rosenkranz. Z. Metallkunde, **49,** No. 6, 316-323 (1958) June.

In thin-walled, intricate die-castings made of the aluminum alloy containing 4.5% zinc, 3.1% magnesium, 0.8% copper and about 0.5% of various other metallic constituents, a structure showing slaty cleavage would develop subsequent to heat treatment. These cracks were intercrystalline and appeared in relatively few grain boundaries, probably in those where a particularly rich precipitate was present, and which were unfavorably positioned relative to the direction of the stress causing rupture. Development of such a slaty cleavage can be avoided by preventing the metal from spontaneous and oriented recrystallization during deformation, and by applying higher temperatures in precipitation heat treatment. Iron, manganese and chromium added in sufficient quantities inhibit recrystallization. Higher precipitation heat treatment temperatures may be applied without loss of mechanical strength, if the metal is permitted to age naturally, between quenching and precipitation heat treatment, for a sufficient length of time. By proper coordination of the factors mentioned one may avoid grain boundary brittleness causing intercrystalline slaty cleavage and also the stress corrosion behavior of the metal can be substantially improved without sacrificing static and dynamic properties of the casting, or the relationship of these properties to one another in various orientations.—ALL. 16583

8.8.5, 8.4.5

Problems Encountered in Casting Reactive Metals. W. A. Aschoff and D. H. Blair. Trans. Am. Foundrymen's Soc., **66,** 257-260 (1958); Modern Castings, **33,** No. 6, 61-64 (1958) June.

Reviews difficulties to be overcome before widespread application of castings of reactive metals (titanium, zirconium, molybdenum, tantalum and niobium). Gas porosity is considered. In vacuum-arc furnace, measurement and control of molten pool temperature is practically impossible. Best answer may be measurement of stream of molten metal as it is poured from crucible by thermopile device; reading depends on emissivity of molten stream. Alloy casting effects are discussed. Zircalloy-2 exhibits as-cast properties equivalent to wrought material with exception of greater ductility. Behavior of superalloys (both iron and nickel base) indicates that they should be classed with the

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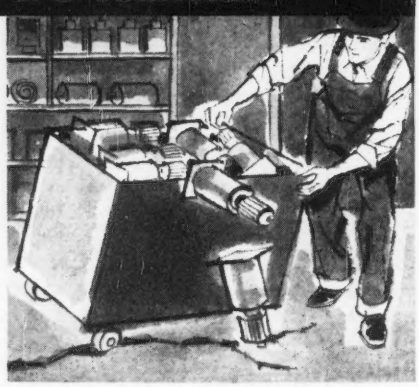

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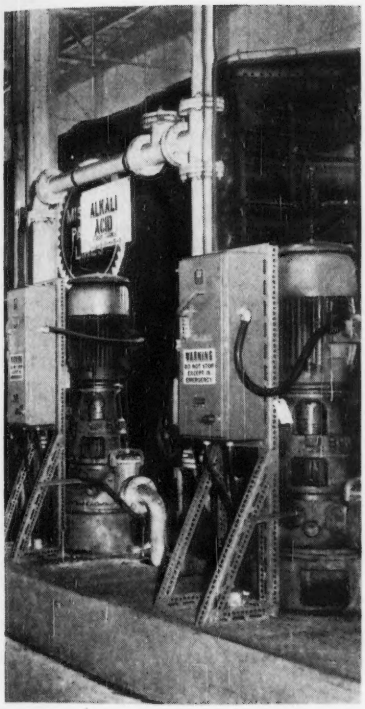

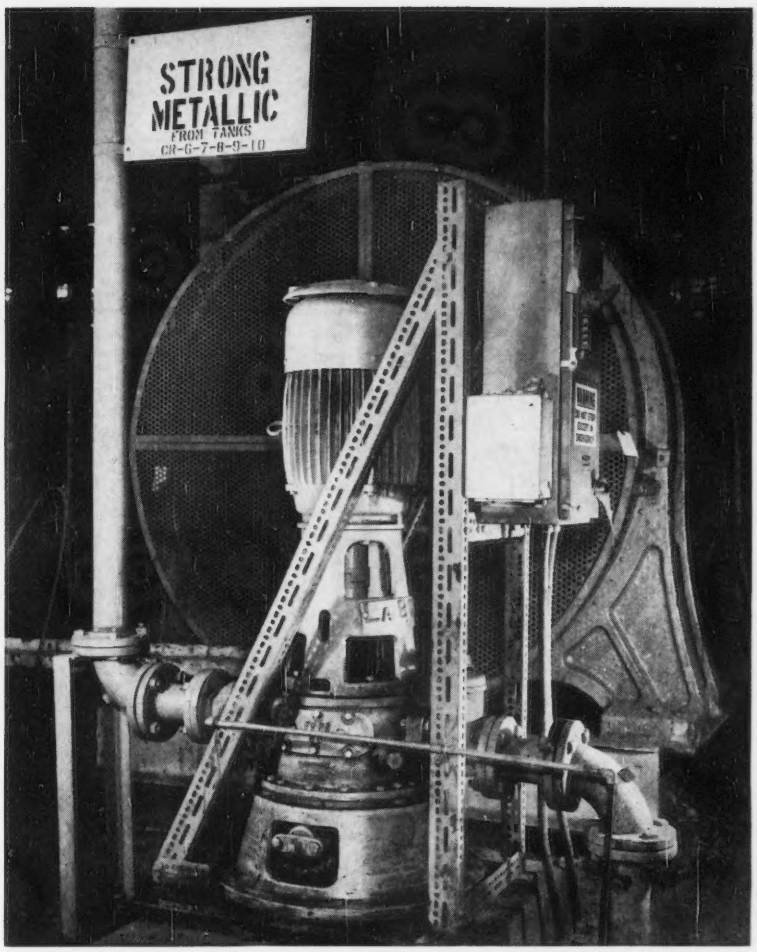

(Photo and information courtesy of *Chemical Processing*)

How cast stainless steel pump parts make corrosive waste recovery more profitable

When pumps can be kept on the go, recovery of chemicals in highly corrosive plating and washing solutions is much more profitable.

And the pumps can be kept going! In a midwest plant treating 1¼ million gallons daily of strong acids, cyanides, and alkaline wash solutions, about 150 type CG vertical pumps...most of them automatic... have been pumping for better than two years. This with practically no time out for maintenance.

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About the alloys

The La Bour Company, Inc. of Elkhart, Indiana, produced the pumps and used its own alloys for the parts described. Both alloys have excellent corrosion and erosion resistance, excellent mechanical properties.

La Bour markets its 9% Nickel stainless casting alloy under the name of "149 Alloy*". It contains, in addition to the Nickel, 14% chromium and 2% silicon.

La Bour designates its 26% Nickel alloy as "Elcomet K*." Besides Nickel, it contains 20% chromium, 3% molybdenum, 2% copper and 2% silicon. This particular combination of elements gives Elcomet K especially good resistance to sulfuric acid and to reducing chemicals and chlorides. It and similar (ACI designation, CN-7M) stainless steel casting alloys have an outstanding record of resistance to metal cleaning and metal plating solutions.

Just out! New booklet!

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8.9 Group 9

8.9.5, 4.4.7

Cargo Space Corrosion in Tanker Ships. (In English.) W. G. Richards. Teknisk Ukeblad, **104,** 869-874 (1957) October 24.

Methods for overcoming severe corrosion of cargo tanks exposed to white or black petroleum products. 16 references.—MR. 16308

8.9.5, 7.5.5, 5.2.2

U.S. Tankers Freed From Corrosion. John Grindrod. Corrosion Technology, **4,** 278-280 (1957) August.

Hulls and ballast tanks of two chemical tankers protected by magnesium anodes.—MR. 14878

8.9.5, 5.2.1, 5.4.5

Corrosion Control in Tankers. A. Logan. Trans. Inst Marine Engrs., **70,** 153-194 (1958) May.

Experience of 135 ships that are cathodically protected or treated with epoxy coatings. 2 plates.—BTR. 16540

8.9.2, 5.11, 5.9.4

Corrosion Prevention in Automobile Bodies. T. P. Hoar. Industrial Finishing, **10,** No. 116, 45-46, 48 (1958).

Corrosion problems associated with car finishing are described; these include unfavorable design factors often leading to premature corrosion of edges, corners, etc. Some remedies are suggested, including some forms of plating or anodizing.—RPI. 16474

8.9.2, 5.8.2

Automotive Parts Cleaners. I. Shafiroff. R. H. Hollingshead Corp. Soap and Chem. Specialties, **34,** No. 5, 57-60, 136-137 (1958) May.

Description of experiments on corrosion and evaporation of automotive parts cleaner fluids. Coupons used in test were of A1 24ST and 4130 steel. Results show that suitable liquid and vapor phase inhibitors for metals encountered in cleaning and adequate buffering action to resist or minimize pH change are major requirements which confront the formulator. Photos, tables. (auth)—INCO. 16654

8.10 Group 10

8.10.2, 6.6.6

Ceramic Investigations of UO_2. Adrian G. Allison and Winston H. Duckworth. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1009, June 16, 1955 (Declassified Feb. 13, 1957), 32 pp. Available from Office of Technical Services, Washington, D. C.

A UO_2 powder with unusually good sinterability was prepared from MCW UO_3 by milling it for 4 hours in 0.02N sulfuric acid or in distilled water and then reducing it to UO_2 in water-saturated hydrogen at temperatures of 930 to 1110 F. Compacts of these powders, pressed at 20,000 psi and sintered at 2800 F. for 1 hour in hydrogen, had bulk densities of 10.75 to 10.93 g./cm³. As-received MCW UO_2 powder was made free flowing by mechanical agglomeration, using camphor as a temporary binder. The agglomerated powders had good pressing characteristics, and the treatment did not appear to affect the sintering behavior or the resistance of sintered compacts to corrosion by 650 F. water or 750 F. steam. Twelve simulated fuel elements were made for inpile tests. Plus 325-mesh MCW UO_2 was hydrostatically pressed into core rods at 100,000 psi and sintered in hydrogen at 3750 F. Bulk densities of the cores varied from 9.71 to 9.96 g/cm³. The sintered UO_2 core were ground to the desired diameter and length, and were clad in Zircaloy-2 tubes by an arc-welding process. Preliminary investigation indicated ceramic extrusion to be a satisfactory method for fabricating UO_2 fuel-element cores, providing that the UO_2 powder used is a readily sinterable type. Using such a powder and a quantity of organic plasticizer that introduced less than 0.2 w/o ash, extrusions 3/16 in. in diameter had bulk densities of from 9.85 to 10.65 g/cm³ after sintering at 3000 F. in hydrogen for 1 hour. The bulk density of compacts made of MCW UO_2 pressed at 100,000 psi and sintered in hydrogen at 3200 F. was increased from 9.50 to 10.38 g/cm³ by milling the as-received UO_2 to a maximum particle size of 5 μ in a rubber-lined mill, using uranium balls. Small lots, up to 3000 g. of UO_2 were fused in an electric-arc furnace using a tungsten-tipped electrode with a water-cooled copper crucible and a protective atmosphere of pure argon. About 5 w/o of the UO_2 was deposited on the cool walls of the furnace by vaporization during the melting operation. About 2000 ppm of tungsten was picked up by the UO_2 during the fusion. Compacts having a sintered bulk density of 10.03 g/cm³ were made by milling the fused oxide to an average particle size of 7.1 μ, hydrostatically pressing at 100,000 psi, and sintering for two hours in hydrogen at 3750 F. Between 0.1 and 1 w/o of zirconium was picked up during milling of the fused UO_2, using a rubber-lined mill and Zircaloy-2 balls. (auth)—NSA. 15248

8.10.2

Corrosion and Metallurgy. (In Czech.) Milic Roubal. Hutnické Listy, **12,** 414-417 (1957) May.

Effects of metallurgical processing on speed of corrosion.—BTR. 15042

8.10.3, 1.2.5

Corrosion of Iron and Steel in the Salt-Crust Treatment of Mine Roads and Its Suppression by Inhibiting Agents. A. L. Godbert and M. C. White. British Safety in Mines, Research, Report 143, October, 1957, 24 pp.

The salt-crust process as means of preventing coal-dust explosions by immobilizing coal dust deposited on mine roads.—MR. 15790

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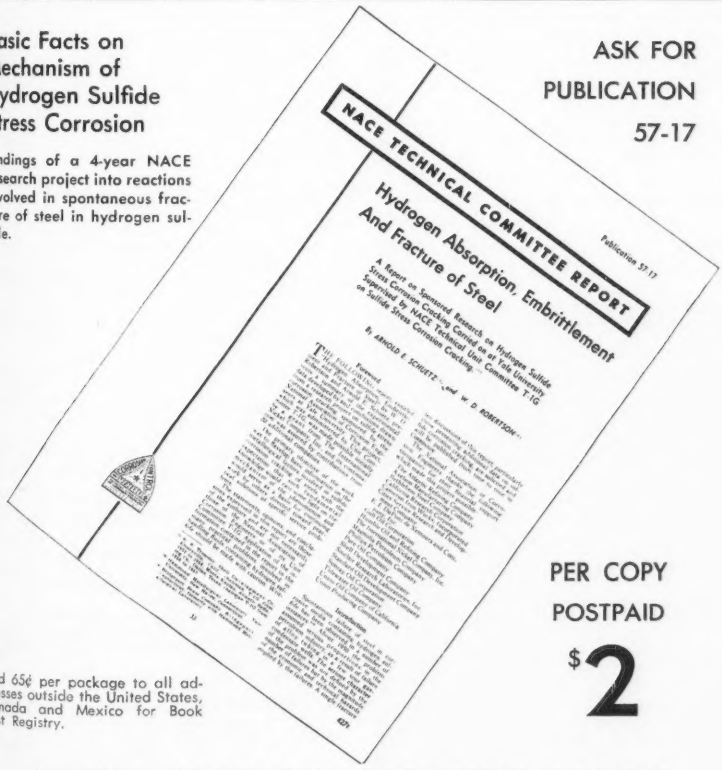

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Industrial Maintenance Painting—Report of Technical Committee T-6D

Corrosion of Zirconium in Hydrochloric Acid at High Temperatures and Pressures, by W. E. Kuhn

Underwater Corrosion of Ten Structural Steels—Part 3, by B. W. Forgeson, C. R. Southwell and A. L. Alexander

Bibliography of Corrosion Products—Ni and Ni Alloys, Cobalt and Cobalt Alloys, Copper and Copper Alloys, Me and Me Alloys, Chromium and Chromium Alloys. Technical Committee T-3B Report, Section 3

Influence of Cold Work on Hydrogen Behavior in Steel, by R. M. Hudson, K. J. Riedy and G. L. Stragand

A Distributed Anode System in an Urban Area, by James P. McArdle, Jr.

Corrosion and Weldability Studies on Chromium-Manganese Austenitic Stainless Steels, by Donald Warren

Corrosometer Plus Statistical Analysis Yield—Unique Approach to Corrosion Problems, by G. E. Moller and J. T. Patrick

Field Service Results of Silicon vs Selenium in Cathodic Protection, by Don P. Wilson

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(b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

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(e) To promote standardization of terminology, techniques, equipment and design in corrosion control.

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An Association of Individuals and Organizations Concerned with Control of Corrosion

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Vol. 16 February, 1960 No. 2

Topic of the Month

Corrosion Design in the Marine Application Of Aluminum Alloy Floodlights*

By R. E. BROOKS*

Abstract

A case history is given of the premature failure of an aluminum floodlight in a marine environment. Design considerations which could have prevented this failure include (1) use of a continuous rubber or other non-absorbent type gasket rather than an absorbent split fiber gasket, (2) use of six rather than four bolts to secure the lens holder to the main body of the assembly, (3) use of anodized aluminum or stainless steel bolts rather than brass or other copper-rich alloy bolts, (4) use of copper-free permanent mold or sand casting alloys such as 360, 43, 356, etc. 5.11

Introduction

MANY ALUMINUM alloys possess excellent corrosion resistance to marine environments. For example, hundreds of aluminum navigation lights have been used successfully on inland and coastal waterways where navigation aids are required. These lights incorporate many of the following proper design factors which contribute to a long service life in a marine or other corrosive environment: (1) all aluminum construction with corrosion resistant alloys, (2) elimination of all possible crevices, especially in critical areas, (3) elimination of bimetallic contacts, (4) faying surface protection and (5) use of non-absorbent gasketing materials. Figure 1 shows an aluminum navigation light with special attention directed to the manner in which a water-tight seal was obtained between lens and lamp housing.

Even though aluminum alloys have been used successfully in many applications similar to the above, there are today fabricators of aluminum products who do not recognize the importance of corrosion design considerations in manufacturing operations. To obtain the optimum performance from aluminum as well as other materials of construction, not only must the proper alloy be used, but also proper design must be utilized. As opposed to the successful use of aluminum navigation lights, the following case history of the premature failure of an aluminum floodlight points out an example where proper design principles were not followed.

Discussion

During early 1959, a corroded aluminum floodlight obtained from a small fishing vessel was examined. The floodlight had been in service for only a short period of time; however, the reflector sheet was so badly corroded that the unit was inoperable.

The complete floodlight assembly consisted of a cast body, a cast lens holder (Figures 2 and 3), a reflector (Figure 4), and a split, absorbent fiber gasket fitted between body and lens holder (Figure 5). The chemical analysis of the reflector sheet is given in Table 1.

Corrosion of the reflector sheet caused failure of the unit. The composition of the reflector sheet meets Aluminum Association Specifications. Failure of the sheet was not due to alloy choice but rather to poor corrosion design. The design considerations contributing to failure of the unit are discussed below.

Design Considerations

The main casting of the floodlight assembly was fabricated so that four raised flats were spaced 90 degrees apart around the periphery of the casting. Holes had been drilled and tapped into each flat. The reflector was then placed into the casting and secured by four brass bolts—thus, water could be entrapped between the casting face and reflector back. A split absorbent fiber gasket was then placed around the periphery of the body with the split in the gasket located at the bottom of the unit.

The final step in assembly was to attach the lens holder to the main casting with four brass bolts. These bolts were so located that the lens holder could not be pulled securely into the main body of the unit (Figure 3). The completed floodlight was then fitted in a cast iron mount which could be placed aboard a vessel to operate in either fresh or salt water—the case in point involved service in a marine environment.

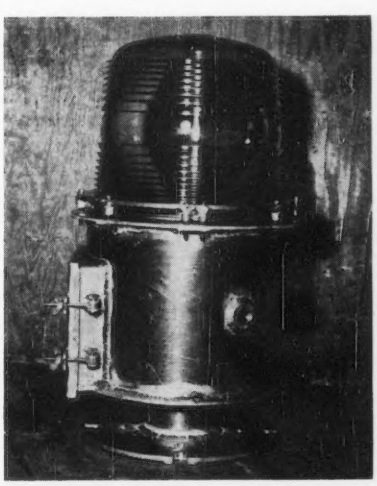


Figure 1—Welded aluminum navigation light of a type used as a navigational aid on practically all inland and coastal waterways.

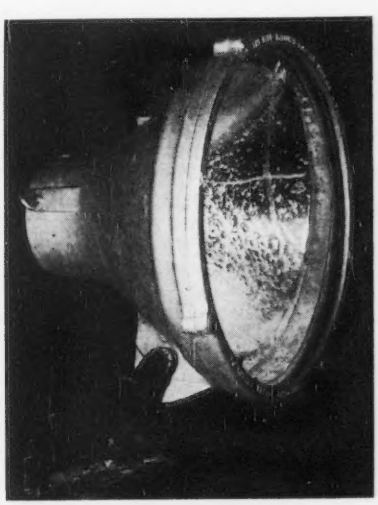


Figure 2—Cast aluminum floodlight.

★ Submitted for publication October 12, 1959.

* Kaiser Aluminum and Chemical Corp., Dept. of Metallurgical Research, Spokane, Washington.

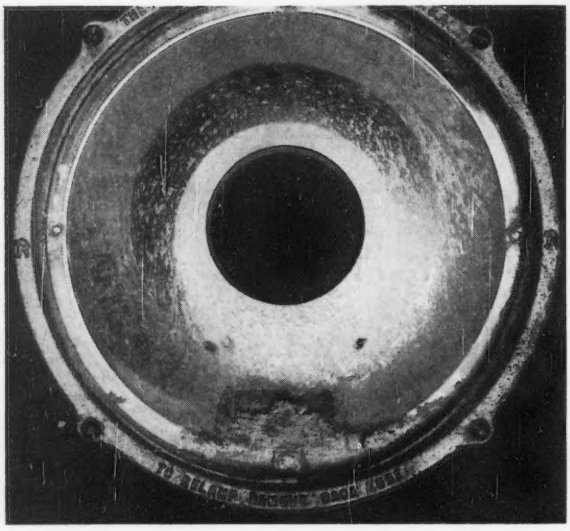


Figure 3—Corroded reflector sheet in place in cast aluminum housing. Severe corrosion has occurred at the bottom and around the brass bolts in the reflector sheet.

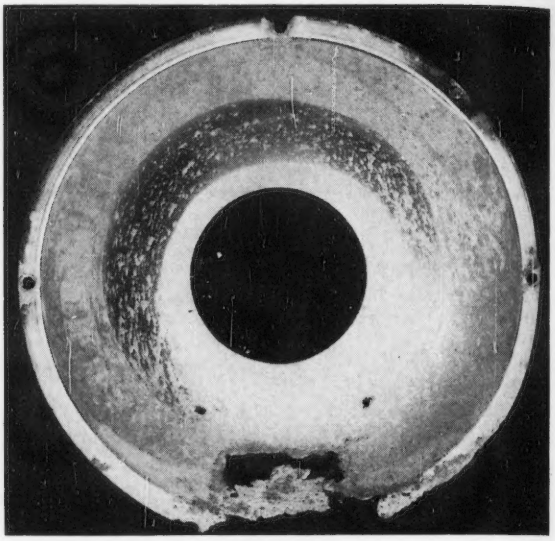


Figure 4—Detail photograph of the corroded reflector sheet.

Figure 5—Split fiber gasket in place in the cast aluminum floodlight. Note the corrosion on the inner surface of the main floodlight body.

TABLE 1—Chemical Analysis of Reflector Sheet

Content, Percent							
Silicon	Iron	Copper	Manganese	Magnesium	Chromium	Titanium	Aluminum
0.10	0.62	0.13	0.003	0.01	0.003	0.004	Remainder

During storms and heavy seas, water collected in the top of the crevice where the cast body and lens holder were joined. The fiber gasket soon became saturated, and water leaked through the split gasket and filled the area both ahead of and behind the reflector sheet. Thus, conditions were ideal for several corrosion processes to function. The processes were as follows: (A) crevice type corrosion between cast body and reflector, (B) galvanic corrosion between the aluminum components and the brass bolts and (C) poultice action where the water saturated fiber gasket contacted the aluminum castings.

Evidence of corrosion resulting from the three processes noted above is as shown: (A) the reflector sheet was completely destroyed at the bottom where in contact with entrapped water, and a loss in reflectivity occurred over the remainder of the sheet where water splash had formed numerous superficially stained areas (Figures 3 and 4), (B) complete destruction of the reflector occurred around the bottom brass bolt as did severe attack of the aluminum at the other bolt holes (Figure 4), and (C) attack of the aluminum casting occurred where in contact with the saturated fiber gasket (Figure 5). All of the above corrosion failures could and should have been prevented by proper design of the unit.

Although not a factor in the failure of the unit, the composition of the castings (Silicon—4.48 percent, Iron—0.30 percent, Copper—2.45 percent, Manganese—0.33 percent, Aluminum—remainder), is not satisfactory for extended service in a marine environment.

Conclusions

To minimize corrosion of the floodlight, the following design considerations should have been observed:

(1) Use of a continuous rubber or other non-absorbent type gasket, rather than an absorbent split fiber gasket.

(2) Use of six, rather than four bolts to secure the lens holder to the main body of the assembly.

(3) Use of anodized aluminum or stainless steel bolts rather than brass or other copper-rich alloy bolts.

(4) Use of copper-free permanent mold or sand casting alloys such as 360, 43, 356, etc.

Only if the proper alloy and design considerations are utilized, as in the case of the aluminum navigation lights and similar applications, will the excellent corrosion resistance of aluminum be realized through a long, trouble-free service life.

Sources of Underground Corrosion Potential Differences*

By K. G. COMPTON

About the Author

K. G. COMPTON has worked with corrosion problems for more than 25 years at the Bell Telephone Laboratories, Murray Hill, N. J. During World War II he was a consultant on the deterioration of materials to the NDRC and the chief of Ordnance; he has also been active on corrosion committees of several technical societies. It was at the State College of Washington that he received his training in electrical and chemical engineering and in electrochemistry.

Abstract

Explains potential of metals in their environments and its role in galvanic corrosion. Sources of potentials discussed include polarization, stray current, dissimilar metals, differences in electrolyte and differences in structure surface. Also discusses behavior of potentials during corrosion. Tabular data and figures given. 3.6.1

Introduction

ALL METALS are considered to have an electrolytic solution pressure or a definite tendency to pass from the atomic to the ionic state when immersed in water. This is opposed by the osmotic pressure of ions in solution which tends to return the metal to atomic form. These two opposing forces at equilibrium determine the potential of a metal to its environment.

If two metals are immersed in electrolytes containing their own ions as shown in Figure 1, where the electrolytic solution pressure P is greater than osmotic pressure p, metal will tend to go into solution as ions, giving up electrons in the process. This will create a negative charge on the metal with respect to the electrolyte adjacent to the metal. On the other hand, if p is the larger, metal ions will tend to go out of solution and cause the metal to become positively charged with respect to the solution.

In both cases a potential difference appears at the interface between the metal and the solution. This potential difference produces what is known as the electric double layer which is said to be one atomic or molecular layer in thickness. Noble metals[1] (e.g., gold, silver, copper) acquired a positive potential; base metals (e.g., zinc, aluminum, lead, iron) acquired a negative potential.

From thermodynamics it can be shown[2] that

$$E = \frac{RT}{ZF} \ln \frac{p}{P} \qquad (1)$$

where R is the gas constant, T is the absolute temperature, Z is the valence of the metal, E is the potential difference across the electric double layer or reversible single electrode potential, p is the osmotic pressure and P the electrolytic solution pressures.

This is the basic Nernst equation. By itself it has little practical significance but is a useful tool in considering the electromotive forces of cells composed of two electrodes immersed in solutions and interconnected by an electrolytic path.

In underground corrosion studies, use is made of a cell composed of the metal electrode under study electrolytically coupled to a copper electrode immersed in a saturated solution of copper sulfate. The potential of such a cell is $E = E' - E_{cu} + X$ where E' is the potential of the metal under study, E_{cu} is the potential of a copper/saturated copper sulfate half cell and X is the sum of the liquid junction potential and the contact potential between metals. The potential measured is

$$E = \frac{0.058}{Z} \log \frac{p}{P} - E_{cu} + X \qquad (2)$$

The effect of concentration on this cell can be shown[3] by substituting kC for the osmotic pressure where C is the effective concentration of the metal ions and k is a constant.

$$E_1 = \frac{0.058}{Z} \log \frac{p_1}{P} = \frac{0.058}{Z} \log \frac{kC_1}{P} \qquad (3)$$

and at concentration C_2

$$E_2 = \frac{0.058}{Z} \log \frac{kC_2}{P} \qquad (4)$$

and by subtracting Equation (3) from Equation (4)

$$E_2 = E_1 + \frac{0.058}{Z} \log \frac{C_2}{C_1}$$

From this relationship, it can be seen that for a ten fold change in the concentration of a bivalent ion such as zinc, a change in potential at the metal liquid interface of 29 millivolts is obtained. Variation in potential with concentration for various metals is shown in Figure 2. When C = 10°, we have the standard electrochemical series for the potentials of metals as given in the usual textbook tables.

Similarly, it can be shown[4] that liquid junction potential between two solutions of different concentration is given by the relation

$$E_x = 2\eta a \frac{0.058}{Z} \log \frac{C_2}{C_1} \qquad (5)$$

where C_2 and C_1 are the effectve ion concentrations on either side of the junc-

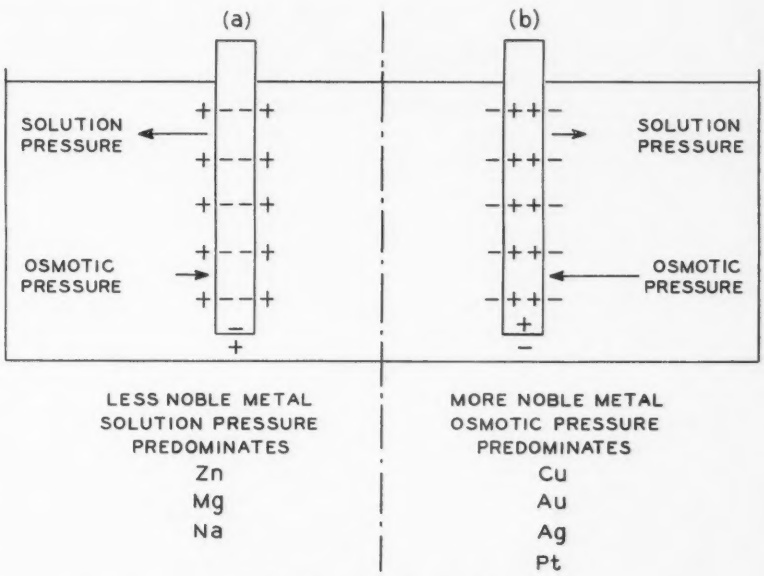


Figure 1—Mechanism of action of voltaic cells.

★ Submitted for publication June 18, 1959. A paper titled "Source of Underground Corrosion Potentials," presented at the Southeast Region Conference, National Association of Corrosion Engineers, Birmingham, Ala., October 11, 1957, and at the 2nd Annual Corrosion Control Short Course and Technical Conference, sponsored by the Miami Section, National Association of Corrosion Engineers, Key Biscayne, Fla., November 14, 1957.

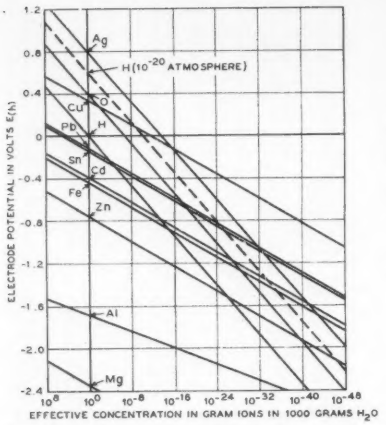


Figure 2—Potential of metals as a function of ion concentration.

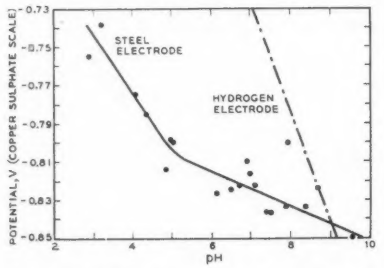


Figure 3—Relation between potential of steel in air-free soils and pH values of the soils.

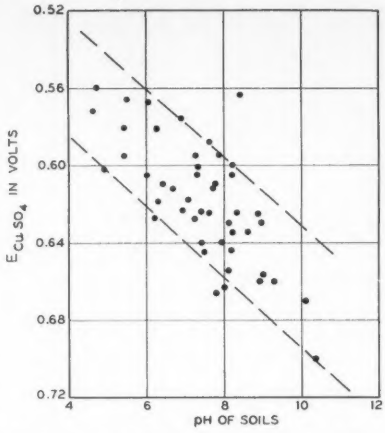


Figure 4—Potential of Pb-Sb cable sheath in soils as a function of pH.

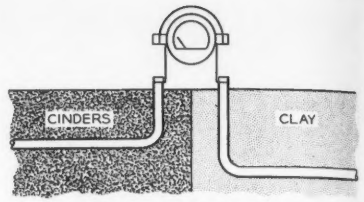


Figure 5—Arrangement of lead pipes in bed of cinders and in clay for measurement of corrosion current.

TABLE 1—Potentials For Metals to Copper-Saturated Copper Sulfate Reference Electrode in Four Soils[7]

Metals	Soil No. 13	23	57	43
pH	9.5	9.4	7.7	3.1
Steel	—0.472	—0.552	—0.602	—0.642
Zinc	—0.692	—0.912	—0.702	—1.082
Copper	—0.152	—0.092	—0.072	—0.142
Lead	—0.472	—0.422	—0.322	—0.342
430 Steel	—0.232	—0.10	—0.052	—0.642
18-8 Steel			—0.062	—0.422
Brass				—0.172

TABLE 2—Potentials for Metal Specimens in Wisconsin Soil

Couple Pos.	Couple Neg.	Potential in Volts	Time
Pb	Steel	0.066	10 sec
		0.205	30 sec
		0.198	5 days
Pb	Zn	0.505	10 sec
		0.500	30 sec
		0.476	5 days
Cu	Pb	0.305	10 sec
		0.370	30 sec
		0.445	5 days
Cu	Steel	0.520	10 sec
		0.530	30 sec
		0.660	5 days
Steel	Zn	0.280	5 days

tion and ηa is the transference number of the anion. This expression is valid only in cases where there is but one species of anion and one species of cation. In other circumstances, additional factors must be introduced into the equation.

Polarization

So far discussion has been limited to the static potentials at the metal-solution interface and at the solution-solution interface. These potentials indicate the source of the energy and the tendency to corrode from galvanic effects. The actual corrosion is dependent upon the flow of current from anodes carrying metal ions into the solution. As corrosion current flows, it produces changes in the electrolyte at both anode and cathode. Anions tend to accumulate at the anode which may make the anolyte more acidic. Cations migrate away from the anode and concentrate at the cathode, rendering the catholyte more alkaline. For the discharge of hydrogen at the cathode an overvoltage is required, and a similar overvoltage is required for the discharge of oxygen at the anode. The shift in potential at a metal solution interface from its steady state open circuit value due to current flow is called "polarization" and is the sum of the effects produced by concentration changes, overvoltage, etc. The effect increases with increased current flow.

Stray Current

The pick up and discharge of stray current, though a serious cause of corrosion of such underground structures as pipelines and cables, is not discussed in this article, though stray currents are a source of underground corrosion potential. These currents can be produced by street railways, interfering cathodic protection systems, unbalanced three-wire Edison direct current systems or ground return telegraph and telephone circuits, etc. Stray currents usually are easy to detect and control measures are well defined. In some cases suitable control measures cannot be applied to existing plant due to a highly complex array of underground structures.

Dissimilar Metals

The increasing use of copper or brass service pipes from the main in the street to the customer's building presents a serious problem to corrosion engineers. The potential between steel pipes and copper tends to exceed 0.5 volts in most soils and that between lead cable sheath and copper is usually greater than 0.35 volts with the copper positive. In some instances bronze valve bodies are attached to steel pipes. Bonding gas and water pipes to power and telephone cable sheaths, though desirable from a safety standpoint, brings these structures under the influence of the copper service pipes.

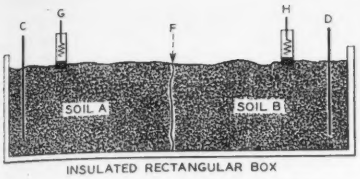
Copper grounds provide another source of lead-copper or steel-copper galvanic couples, and vary from an individual copper clad steel rod to elaborate heavy copper wire grids. In some instances, a buried bare copper wire acting as ground and neutral is included with the power line or feeder. In d-c systems, an unbalance to this neutral can produce stray current in addition to the galvanic couple effect. In multi-grounded systems, a copper ground is placed at the base of each pole.

The couple between bright lead sheath and bright steel is not particularly harmful to steel because hydrogen overvoltage on lead is high. Potential of the lead-steel couple is about 0.2 volts as compared to 0.5 volts for lead-copper. Combination of high overvoltage and low potential difference would tend to minimize the corrosion current.

If steel is in soil of one pH and lead in soil of another from Figures 3[5] and 4[6] it is apparent that the potentials could vary from steel 0.3 volts negative to almost zero. The flow of current would make the anode environment more acidic and that of the cathode more alkaline, which would tend to pull the potentials together.

Table 1 gives potentials for several metals to the copper/saturated copper sulfate reference electrode in four soils.[7] The analytical information on the soils is given in the original publication. These data illustrate the variations in the relative potentials of metals in different soils. Similar data are given in Table 2 for several combinations of metal specimens in a Wisconsin soil. The author made no effort to analyze the soil but did record the pH and resistivity which were 8.4 and 1180 ohm-centimeters, respectively. A comparison of data in Figures 3 and 4 and Tables 1 and 2 reveals a wide variation in the reported potentials and is typical of data in this field.

A frequent source of corrosion potential results from cinder fills containing



INSULATED RECTANGULAR BOX

C & D STRIPS OF LEAD CABLE SHEATH

G & H COPPER/COPPER SULFATE REFERENCE ELECTRODES

F BOUNDARY BETWEEN SOILS

Figure 6—Device for soil potential studies.

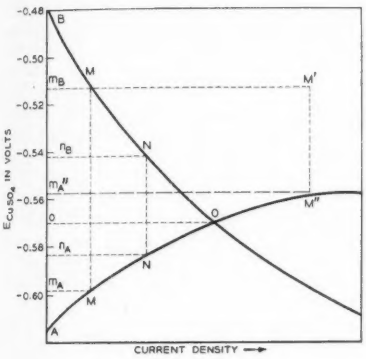


Figure 7—Polarization diagram.

a significant amount of unburned coal or coke in contact with underground metallic structures. The potential of carbon or graphite as used for anodes in ground beds is about 0.6 volts positive and to lead and 0.8 volts positive to steel as measured in a neutral California soil. Robson and Taylor[8] conducted experiments with combinations of clay and cinder fills. They buried lengths of lead pipe in a bed of cinders in clay soil and a second set directly in the clay soil as shown in Figure 5. They measured the corrosion current, potential and the loss in weight of the section of pipe in the clay. Results obtained are given in Table 3.

Differences in Electrolyte

Many cases of corrosion result from potential differences associated with differences in the electrolyte in contact with the underground structure. These may be differences in pH, concentration of ion species, dissolved oxygen, kind of ion species or combinations of these. For example, the data in Figures 3 and 4 suggest significant differences in potential between portions of an underground structure of lead or steel which are in contact with soils of different pH. This can be illustrated by an experiment in which several soil combinations were placed in a soil box as illustrated in Figure 6. Measurements were made of the potentials between the reference electrode and the lead specimens in their respective soils, C-G, and D-H; the liquid junction potentials at F, G and H and the potential between lead specimen C and lead specimen D. The combinations are shown in Table 4 and the data are given in Table 5.

TABLE 3—Effect of Various Treatments To Pipes in Terms of Mean Daily Reading of Current*

Unit No.	Method of Pipe Protection in Cinders	Daily Current Readings Expressed as M. Amp.		
		Maximum	Minimum	Calculated Average
1	No treatment	9.2	1.1	3.69
2	Woven Glass and Grease	4.7	Nil	1.70
3	Clay	Nil	—0.4	—0.10
4	PVC Tape	Nil	Nil	Nil
5	Bitumen Paint	10.0	3.0	5.32
6	No Treatment	13.6	2.1	6.74
7	Sand	Nil	—0.5	—0.10
8	Polythene Tape	Nil	Nil	Nil
9	Chalk	Nil	—0.1	Nil
10	Red Lead Paint	12.9	2.1	5.90

* Duration of test in all cases 337 days.

TABLE 4—Voltage, pH and Resistivity of Soil Combinations (See Figure 6)

COMBINATION 1—EXTREMES OF pH			
NP5	pH 10.1	E = — 0.670V	Res 480 Ohm—CM
Buf 13	pH 4.6	E = — 0.572V	Res 22,000 Ohm—CM
COMBINATION 2—EXTREMES OF POTENTIAL			
SB1	pH 7.8	E = — 0.666V	Res 3800 Ohm—CM
Buf 9	pH 4.7	E = — 0.560V	Res 13,300 Ohm—CM
COMBINATION 3—NEUTRAL BUT EXTREMES OF E			
Wis 2	pH 7.4	E = — 0.640	Res 1920 Ohm—CM
Md 2	pH 7.2	E = — 0.595	Res 1000 Ohm—CM
Buf 1	pH 8.2	E = — 0.635	Res 1680 Ohm—CM
Buf 3	pH 8.3	E = — 0.625	Res 9000 Ohm—CM

TABLE 5—Potentials Measured in Soils (See Figure 6)

Soil A	Soil B	E_{C-D} Volts	E_{G-H} Volts	E_{C-G} Volts	E_{D-H} Volts	I_{C-D} Milliamps
1. Np	Buf 13	—0.175	—0.033	—0.698	—0.557	.....
		0	0.0195	—0.636	—0.616	0.055
After 30 Minutes		—0.112	—0.038	—0.653	—0.575	.....
2. Sb 1	Buf 9	—0.108	—0.0138	—0.645	—0.514	.....
3. Md 2	Wis 2	0.118	0.018	—0.446	—0.538	.....
4. Buf 1	Buf 3	0.043	—0.006	—0.661	—0.649	.....

TABLE 6—Potentials Obtained Between Filmed Specimen and Freshly Abraded Specimen

	TIME IN MINUTES						
	0	30	60	90	120	0	60
Potential in Volts*	.173	.150	.148	.148	Shorted	340	164

* Freshly abraded specimen was negative to filmed specimen.

The data in Table 5, in addition to revealing potentials between similar metal electrodes in excess of 100 millivolts, also show a source of error in making measurements with reference electrodes such as the copper/saturated copper sulfate half cell. The potentials G-H are liquid junction potentials which could be encountered in placing two half cells in different positions in contact with the earth. This substantiates the data in another article.[9]

Variations in oxygen concentration in soils or soil waters have a combined effect on corrosion. Potentials of the metal electrodes vary with variations in dissolved oxygen and the depolarizing effect of oxygen on the hydrogen at the cathode is of considerable significance. To illustrate the former case, two specimens of lead cable sheath were placed in beakers of San Francisco manhole water. Water in the beakers was connected electrolytically by wet strips of blotting

paper. Measurements were made of sheath specimen potentials after bubbling O_2 through water in one and N_2 through the other. Specimen in the oxygen saturated atmosphere reached a potential of —0.461 volts to a copper sulfate electrode and that in N_2 indicated a potential of —0.585 volts. Potential between the two specimens checked this difference by 0.126 volts with the oxygen rich specimen positive.

To illustrate the depolarizing effect of oxygen on the hydrogen evolved at the cathode, a specimen of lead cable sheath was placed in a slurry of salt marsh soil and polarized cathodically. The current density required to maintain the polarized potential at —1.50 volts to a copper sulfate half cell was between six and eight times greater when oxygen was bubbled through the slurry than when nitrogen was used. This effect tends to keep the cathodic areas of a corrosion cell at a more positive potential by reducing the hydrogen and lowering the polarization.

Differences in ion species at two points on the surface of an underground structure will cause differences in potential. The magnitude of the difference in potential will depend upon the particular ion species present and the concentration. However, an example of this difference can be seen in the experiment in which two specimens of lead cable sheath alloy were placed in beakers of sea water and manhole water which were connected electrolytically by strips of moist paper. The specimen in sea water was 50 to 60 millivolts positive to that in manhole water.

Similarly, variations in concentration of a particular set of ions produce differences in the potential of metal surfaces. If the variation is only that of metal ion concentration, the potential will become more positive by $\frac{0.058}{Z}$ volts for each tenfold increase in ion concentration. In other cases, differences are obtained which may be of a different magnitude. If two lead sheath specimens are placed in sea water diluted 100 to 1, a potential of 30 millivolts is obtained, with the lead in the diluted sea water negative or anodic.

Differences in Structure Surface

One of the major differences in the surface of a structure is the presence or absence of an oxide or other film. In the case of lead, the difference between a filmed lead surface and one which is bright and freshly abraded can exceed 0.200 volts when in contact with soil. In one experiment with cable sheath in contact with Westchester soil, potentials given in Table 6 were obtained between a filmed specimen and a freshly abraded one. The latter was negative to the filmed specimen.

Mill scale on steel is cathodic to steel by a potential of 0.175 to 0.200 volts depending upon the electrolyte to which the combination is exposed. A new pipeline or lead sheathed cable placed underground often will suffer corrosion at a highly accelerated rate as a result of bonding it to an older and well filmed pipe or cable due to the potential differences.

Scratches on cable or pipe surfaces placed underground will provide anodic areas of bare metal. Pit and crevice bottoms will be anodic due to differential aeration. Similarly, the under side of a pipe or cable usually will be anodic to the upper side due to differential aeration as areas will be under pebbles or other insulating foreign bodies in contact with the structure.

Observed Potentials During Corrosion

Polarization curves are given in Figure 7 for the situation described in Table 6. In a particular experiment, the potential of the filmed or cathodic lead was —0.48 volts as shown at B, and the freshly abraded lead was —0.625 volts as shown at A. When short circuited through a zero resistance milliammeter and held close together, the current was 55 microamperes for 2 square inches of exposed surface, and the potential of the shorted couple to a copper sulfate electrode was —0.57 volts.

In Figure 7, circuit resistance is assumed to be zero and point 0 is the potential of the corroding couple (where the areas are equal). If the resistance through the electrolyte is not zero, an RI drop such as N-N or M-M occurs in the solution and the potentials at the anode or cathode surface becomes n_A or m_A and n_B or m_B respectively. These potentials could be measured by bringing the tip of a small exploring reference electrode close to the anode or cathode surface. If the relative area of the anode is reduced, its current density might be M″ and its potential m''_A.

Any measurement between a reference electrode and a corroding couple is likely to produce potential values that are the resultant of the anode's polarized potential and the cathode and the RI drop in the electrolyte. These values will vary with the location of the reference electrode with respect to the anodes or cathodes. The closer the reference electrode is to the anode or cathode, the more nearly the measured potential will approach the true values.

In addition to variations due to position, potentials will vary with time because of changes in concentration, variations in dissolved oxygen, films formed on the electrodes and variation of polarization with time. Most investigations indicate whether the reported potential is the initial potential, the steady-state potential or the potential after a specific interval.

Conclusions

The sources of potential in underground corrosion are many and varied. Large scale couples can be detected by electrical measurements; small local couples cannot be detected. By proper use of cathodic protection, most corrosion couples can be nullified. The sources of potential for local couples usually are differential aeration, inhomogeneities of metal surface and dissimilar metals in contact.

The sources of large scale or long cell couple potentials can be (1) dissimilar metals such as copper-iron or copper-steel, (2) cinder fills (which provide such couples as carbon-iron or carbon-lead, (3) concentration differences, (4) differences in alkalinity and (5) differences in ion species.

In addition, the interference currents from neighboring cathodic protection systems and stray currents from ground return circuits or electric railways must be considered. These should be eliminated before searching for the other sources of corrosion potential.

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Any discussion of this article not published above will appear in the December, 1960 issue.

Anodic Passivation Studies*

By J. D. SUDBURY, O. L. RIGGS, JR., and D. A. SHOCK

Abstract

A brief review of the present theories of passivity indicates that corrosion can be completely stopped by formation of a very thin oxygen containing film. This concept has not been previously used in practical control of corrosion. Using a potentiostat for maintaining a constant potential of a metal to be protected, many variables involved in the anodic control of corrosion were studied. This technique is shown to be useful in HNO_3, H_2SO_4, H_3PO_4, NH_4NO_3, Al_2SO_4, NaOH and numerous other oxidizing environments being handled in mild and stainless steels. Anodic protection was possible in acid-base neutralizations and acid-hydrocarbon reactions so long as adequate conductivity is maintained. Control has been established at temperatures as high as 500 F. Electrical measurements show the film resistance to be in the order of 10^4-10^6 ohms/-cm^2. This extremely high resistance allows establishment of protection in very complex geometrical vessels, and even in flow lines.

A major achievement presented by this report is the development of a potential controller which allows metal passivation to become of immediate practical importance. Pilot studies using a 500 gallon 304 stainless vessel filled with 67 percent H_2SO_4 showed that corrosion could be completely stifled for indefinite periods by application of almost negligible currents. It is possible by laboratory studies to predict the current and voltage requirements for anodically protecting process or storage vessels. These requirements are summarized for numerous systems.

3.8.3

Introduction

WHEN THE surface of a metal has been rendered inactive to its environment, it is said to be passive. This phenomenon is readily illustrated by such things as: (1) the inactivity of mild steel in concentrated nitric acid, and (2) the inactivity of aluminum in the atmosphere. This subject has long been of interest to the electrochemist who has studied the effect of current on the potential behavior of a steel anode (anodic polarization studies).

The phenomenon can best be described by examination of typical polarization curves (Figure 1). Curve A is the result of measuring the potential as the current through the anode is increased. If the experiment is run so that currents are measured as the potential is shifted, Curve B results. A further examination of Curve B describes the phenomenon of passivity.

As polarization begins the major reaction is:

$$Fe \rightarrow Fe^{++} + 2e^-$$

as illustrated by *a* of the curves A and B. As the potential shifts in the more noble direction, a critical value *b* is reached on curve B at which the current suddenly decreases. This point has been designated as the Flade Potential. Portion *c* of the curve B defines the passive region in which potential shifts of as much as 1 volt may occur with very low applied currents. The anode reactions in this region change to:

$$Fe \rightarrow Fe^{+++} + 3e^-$$

At point *d*, the current begins to rise rapidly with further slight potential shifts as the system is no longer passive. The anodic dissolution of iron occurs rapidly. This is undoubtedly due to some change in the electrode reaction.

In order to maintain a piece of metal passive, it is necessary only to maintain its potential within the passive range as defined by this curve. The equipment used most successfully in this work was designed by Hickling[1] who designated this equipment as a potentiostat. Much work has been done unsuccessfully in an attempt to make this technique practical on a commercial basis. Edeleanu[2,3] and Berwick[4] have come close but have concluded that instrumentation is not yet available to maintain the desired potential when very large currents are required for industrial applications.

The anodic polarization studies presented in these pages summarize work done in further development of this concept. Comprehensive data were taken to allow for a better definition of systems where anodic protection is theoretically possible. Development of an inexpensive, high current capacity potential controller made this technique of immediate value in the control of chemical process, and storage problems caused by corrosive liquids. This work also has led to an improved understanding of the nature of the protective barrier and the mechanism of passivity.

Theories of Passivity

The mechanism of passivity has been a subject of much debate for many years. In September, 1957, an international conference[5] was held to discuss the latest thinking on the subject. It was generally agreed that several conditions must be met for passivity to occur. These include:

1. The protective film must be less than 100 Å thick.
2. The film must require no more than 10^{-2} coulombs/cm^2 to be formed.
3. The film must be an electronic conductor.
4. The E vs I characteristics defined by Figure 1 must be satisfied.
5. The film must be formed very rapidly.
6. The passivated metal must be oxidized.
7. The passive film must be hydrophobic.
8. The passive film must be relatively acid insoluble.

Beyond this agreement there is a considerable divergence of opinion as to the exact nature of the passive layer. No attempt will be made to evaluate all the work which has been done. However, the theories can be put in three broad categories.

1. The oxide film theory proposes that iron dissolves and then reacts with oxygen to form protective precipitates

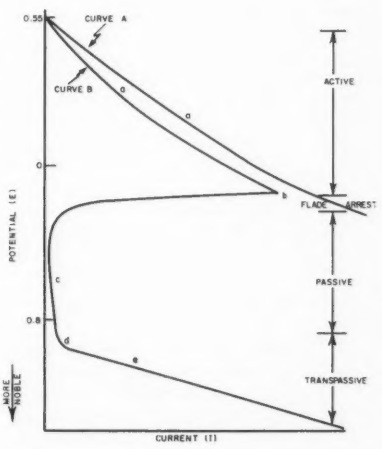

Figure 1—Anodic polarization curves for steel in sulfuric acid. Calomel reference electrode.

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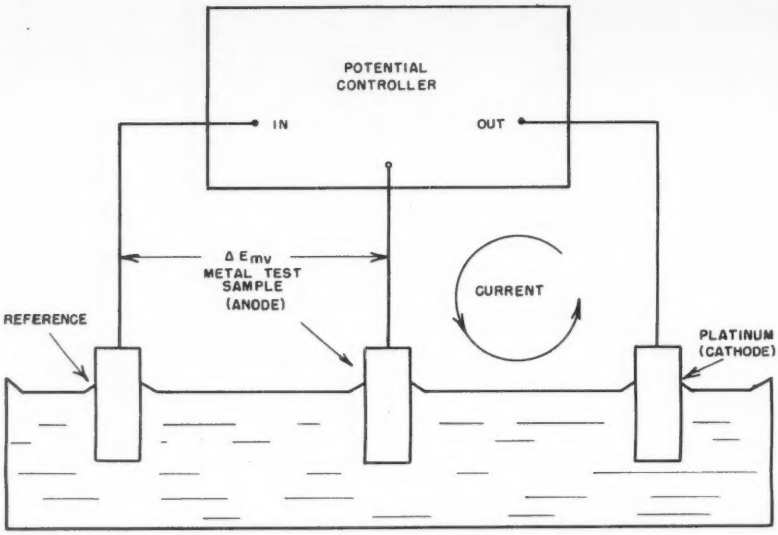

Figure 2—Scheme of electrodes-potential-corrosion rate-controller position.

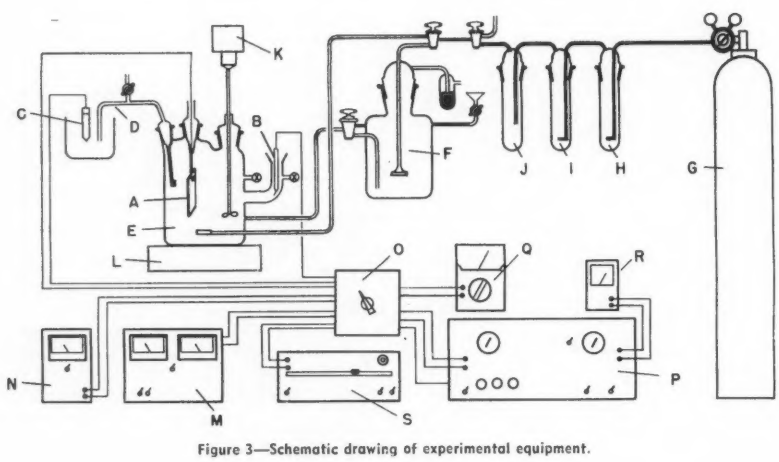

Figure 3—Schematic drawing of experimental equipment.

of iron oxide.[6-19] The film provides a chemical and physical barrier.

2. Proponents of the adsorption theory propose that approximately a monolayer of adsorbed oxygen causes passivity.[20, 21]

3. At above mentioned conference, Hackerman[22] presented his sequence model theory. This concept requires the initial adsorption of oxygen followed by the slow development of an amorphous iron-oxygen structure. This occurs as iron migrates from the base metal into the adsorbed oxygen film. This theory received appreciable support in other papers.[23-25]

Experimental

All of the experiments described in this report use the same basic circuit as shown in Figure 2. This circuit consists of:

1. Anode used as test specimen.

2. Inert cathode—usually platinum.

3. Reference electrode—any standard half-cell such as calomel, silver chloride or hydrogen cell.

4. Potential controller—maintains any preset potential of the anode with respect to the reference electrode. The micro current flow between reference and anode acts as a signal to the controller to alter the current flow between anode and cathode.

Corrosion Test System

A schematic drawing of the experimental setup is shown in Figure 3. The specimen which is to be anodically polarized is assembled at position (A). The platinum electrode (B) and the saturated calomel reference electrode (C), complete the electrochemical circuit. The calomel electrode and metal specimen are connected by an agar-KCl bridge (D). The corrosion test chamber (E) is filled with the selected corrosive fluid which has been properly treated (aerated, deaerated, chemical reagent added, etc.) in the solution preparation cell (F). The gas (G) flows through a series of scrubbing bottles. The first bottle (H) contains 1 percent KOH. The second bottle (I) is filled with distilled water. The third bottle (J) serves only as a trap.

The test solution is stirred by use of an air driven motor and glass rod stirrer (K). Various elevated temperatures are reached by use of the heater (L) below the corrosion test chamber. The electronic portion of the experimental equipment is comprised of power supply (M), voltmeter (N), a junction-switch box (O) for all electrical leads (serves to switch in or out of the circuit either the power supply or the potentiostat), the potentiostat (P), a vacuum tube voltmeter (Q), a microampere meter (R), and a recorder (S). The detailed experimental work was done by using a potentiostat design similar to one used by Hickling.[26] A similar device is now being manufactured in England.*

Test Specimens

Each of the metals studied (listed in Table 1 along with elemental analysis) was milled into ⅜ inch by 3 inch (1 cm × 7.6 cm) strips, referred to as corrosion test coupons. Metals used included 302, 304, 310, 316, 405, 410 and 446 stainless steels, 1020 mild steel, titanium, aluminum and bronze. The surface of each coupon was cleaned by degreasing in toluene, rinsing in isopropyl alcohol, and drying in the air. In order to make the surface active immediately upon exposure to the corrosive environment, the coupon was electrolytically descaled in 67 percent by weight H_2SO_4.

Procedure for Passivation

Electronic instruments required at least 15 minutes for warmup. During this period the corrosive acid and the metal coupon were placed in the test chamber. The potential of the metal with respect to saturated calomel electrode was measured to be certain that the metal surface was in an active condition. For example, 316 stainless steel in 67 percent sulfuric acid has a potential of 360 mv when compared to the calomel. However, current to exceed the Flade potential and thus passivate the metal is applied by use of the potentiostat or a manually controlled external power supply. After passivity is achieved the passive potential is maintained automatically by the potentiostat.

The conventional potentiostat was used for most of the laboratory work. Where surface areas larger than a few square centimeters are involved, this device is not capable of supplying adequate current to maintain the metal passive. In order to apply this passivation technique to large scale processes and storage vessels it has been necessary to develop a potential controller capable of supplying very large currents (up to several hundred amperes).

The authors' work has led to the de-

* Shandon Automation and Electronics, Ltd., 6 Cromwell Place, London, S.W. 7, England.

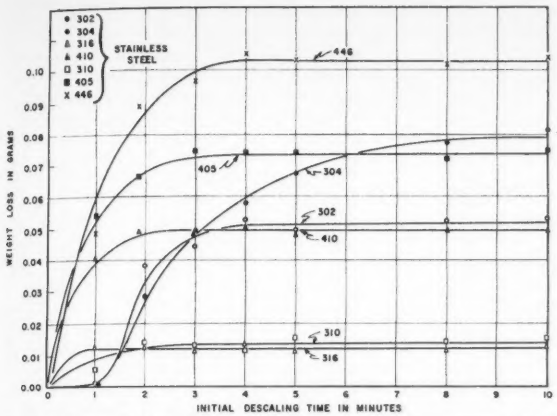

Figure 4—Effect of descaling time on metal loss of coupons exposed in 67 percent by weight sulfuric acid for one hour at 75 F.

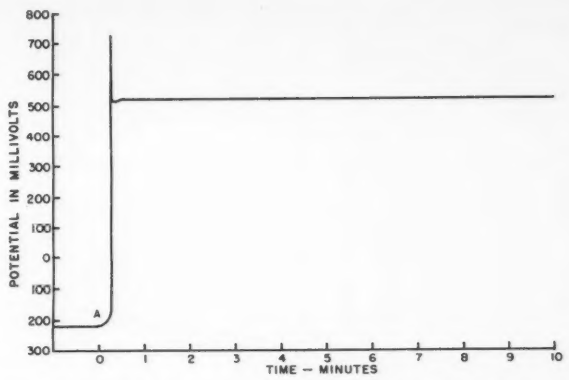

Figure 5—Potential shift during passivation of 316 stainless steel in 67 percent sulfuric acid. Calomel reference electrode.

velopment of an anodic protection system which permits the controlling of corrosion in large vessels. The purpose of this paper will not be to describe the system in detail but will instead describe the results which are possible through its use. The two succeeding papers emphasize the type of large scale operations where this system is applicable.

Results and Discussion

Most of the results presented in this report were obtained using 67 percent H_2SO_4. Similar data can readily be taken for other systems of interest.

Effect of Surface Preparation

To obtain accurate, reproducible potentials, the stainless steel coupon should have an active surface prior to exposure in test system. This active surface was obtained by electrolytic descaling using 1.5 amps for a 17 cm^2 coupon (85 ma/cm^2). In an effort to establish the proper time duration for descaling a given type, a series of descaling times from 1 minute to 10 minutes was used. At each time interval, the coupons were then exposed for one hour to 67 percent H_2SO_4 as shown in Figure 4. From these data it was concluded that the safest descaling period was approximately 10 minutes. The metal specimen would then have a reproducible active potential. For example, 316 stainless steel coupons gave a potential reading of 360 ± 10mv. These data also begin to show the relative corrosion resistance of the various types of stainless steel.

Potential Shift During Passivation

As anodic current is passed through coupons, a shift in potential in the more noble direction occurs. An increased current with a resultant greater potential is reflected by increased metal loss. This increased corrosion rate continues until the Flade Potential is reached. At this time a sudden reduction of metal dissolution and a pronounced potential shift occurs in the more noble direction. This potential shift can be recorded as shown in Figure 5 for 316 stainless in 67 percent H_2SO_4 at 75 F. This very abrupt shift occurs as soon as the required current is introduced by the potentiostat at point A on the time axis. The shift of 900 mv is typical of most steels in acid. The potentiostat maintains this passive potential very accurately for as long as desired.

In some cases the potential shift is less abrupt when the passivating current is applied. In Figure 6 the record made by stainless steel in high purity water to a pH of 9.5 with LiOH is shown. A potential shift to the preset value of 320 mv occurs only after 10 minutes. This slow response seems to be characteristic of other bases such as NaOH.

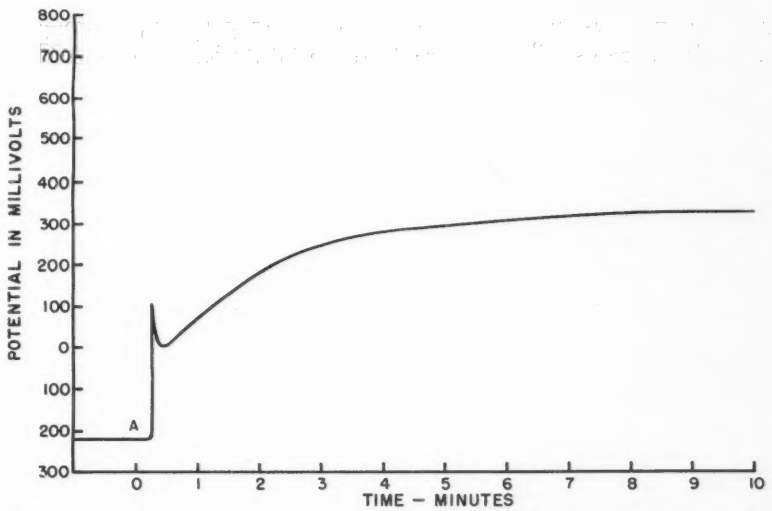

Figure 6—Potential shift during passivation of 316 stainless steel in LiOH at pH 9.5. Calomel reference electrode.

TABLE 1—Current Density Required to Maintain Passivity or Various Metals Calculation of Apparent Resistance of Passive Layer

Metal Type	Current Density, Microamps per cm^2	Potential Between Anode and Cathode	Total Film Resistance, Ohms per cm^2
302	2.2	—1,100 mv.	500,000
304	3.8	— 850	224,000
310	0.5	—1,050	2,100,000
316	0.1	—1,750	17,500,000
405	2.7	— 900	333,330
410	10.9	—1,100	100,900
446	0.7	—1,250	1,786,000
Mild Steel	15.0	— 380	26,000
Titanium	0.08	—1,400	17,500,000
Hastelloy B	0.5	90	180,000
Carpenter 20	0.03	—1,400	46,000,000

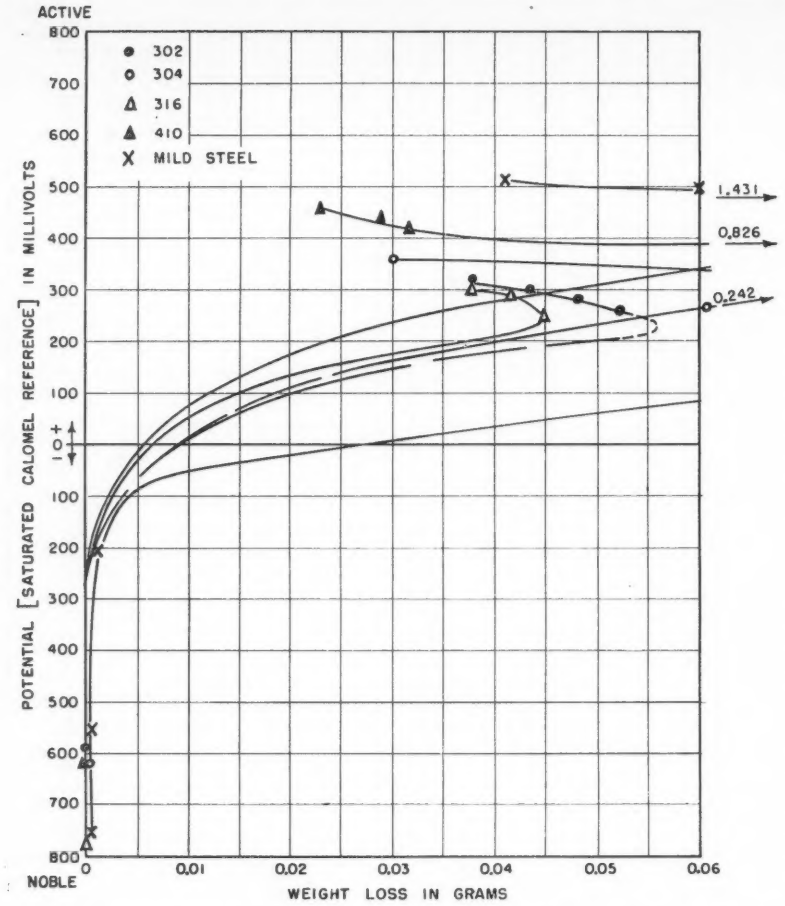

Figure 7—Effect of potential on the weight loss of stainless steel exposed one hour to 67 percent by weight sulfuric acid at 75 F.

In Figure 7, the effect of this potential shift in reducing corrosion is illustrated. Until passivity is achieved the corrosion rate rapidly increases. At passive potentials, corrosion rates are negligible. In all cases when the passive potential was purposefully exceeded, an accelerated anodic dissolution of metal began. It was readily detectable by discoloration of metal and solution (yellow or green).

Current Density Requirements

When a critical current is reached at the Flade potential, the anodic dissolution of metal stops as the potential reaches the passive region. A small current is required to maintain this condition. Very accurate determination of the current required to establish and maintain protection is possible. Actually, the current requirements are somewhat time dependent so a measurement of total charge per unit area to establish passivity is probably more significant than current density alone. However, for ease of measurement, a curve such as in Figure 8 is usually determined for each system studied. The current to establish and to maintain passivity of 316 stainless steel in 67 percent H_2SO_4 is indicated on this curve.

Current requirements have been measured under many different conditions.

Effect of H_2SO_4 Concentration. It is well known that the corrosivity of H_2SO_4 reaches a maximum in the concentration range at about 55 percent by weight. These data are confirmed by Figure 9, which summarizes the weight loss of a 0.5 × 3 × 0.032" 316 stainless steel coupon immersed for 1 hour in varying concentrations of H_2SO_4. Very little metal loss occurred at either 1 or 105 percent H_2SO_4. The current density required to establish passivity was determined at each concentration. Unlike the corrosion curve, the current density required to passivate 316 stainless decreased with increasing concentration of H_2SO_4. In 105 percent H_2SO_4 (20 percent oleum) almost no current was required to obtain the passive state.

Effect of Surface Area. It was desirable to know whether current requirements determined on small surface area specimens could be extrapolated to predict current requirements for large vessels. This point was examined by using a series of 304 stainless steel beakers of identical shape, and of increasing surface area. The currents required to establish and maintain passivity were measured and plotted in Figure 10. In each case passivity was achieved in about 3 seconds. To achieve passivity required about 14-15 × 10^{-3} coulombs/cm².

Based on these data it is readily seen that current density for passivation is independent of total metal surface area. This allows extrapolation of laboratory data to field installations. As will be shown later, it is possible to reduce the current required to passivate a large vessel if one can wait to establish protection.

Effect of Temperature. The effect of temperature on the current required to maintain passivity is shown in Figure 11 for 302, 304 and 316 stainless in 67 percent H_2SO_4 up to 200 F. The current requirements increase rapidly over the entire range. The increase is much more rapid above 150 F. This must in someway be related to an increased solubility of the passive layer at elevated temperatures.

Effect of Different Metals. Attempts have been made to passivate a variety of metals in 67 percent H_2SO_4. Metals successfully passivated are listed in Table 1. Apparently all of the ferrous metals can be handled successfully. Copper base metals such as brass, bronze, aluminum-bronze, etc., could not be passivated. Rare metals (Ti, Zr, Hf, Nb) used to promote corrosion resistance in special systems can all be passivated. Current requirements vary widely with these different alloys. The 316 stainless requires an especially low current to maintain passivity.

The potential across anode and cathode of polarization set up is recorded in column 3 so that an apparent resistance of the passive film can be calculated using Ohm's law. It is seen that resistances are very high—ranging from 26,000 ohms/cm² for mild steel to 46,000,000 ohms/cm² for Carpenter 20. As will be shown later these high resistances ensure maximum throwing power of anodic protection.

Effect of Different Chemical Environments. Most of the work reported here has been done using 67 percent sulfuric acid; however, exploratory work has been done to give some indication of how widely applicable anodic protection may be. The basic E vs I curves have been determined for several systems. Based on this curve, the curent density to establish and to maintain passivity can be estimated. The current densities to establish and maintain passivity in a variety of systems will be summarized in a later paper. Based on these data, it is apparent that passivity can be established in a broad variety of chemical environments, including phosphoric acid, nitric acid, sodium hydroxide, lithium hydroxide, ammonium nitrate and aluminum sulfate.

It was shown that it was impossible to establish passivity in reducing systems such as HCl, NaCl, $FeCl_2$ $SnCl_2$, $HI \cdot H_2O$. Although these systems could not be passivated it was shown that

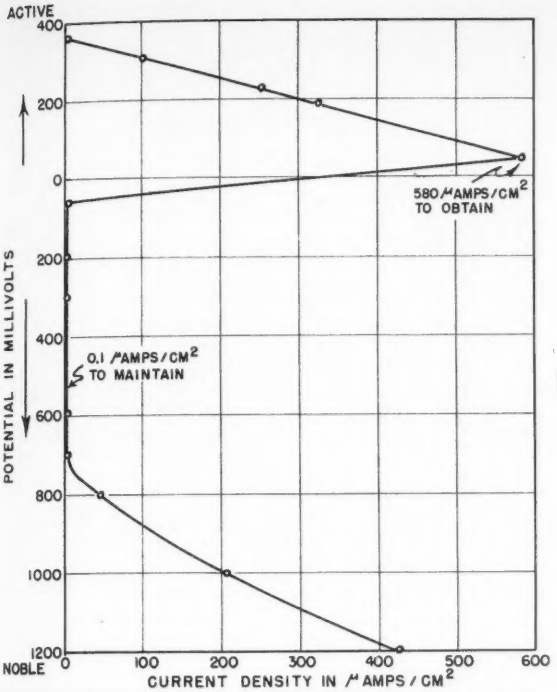

Figure 8—Anodic current required to passivate 316 stainless steel in 67 percent sulfuric acid at 75 F. Calomel reference electrode.

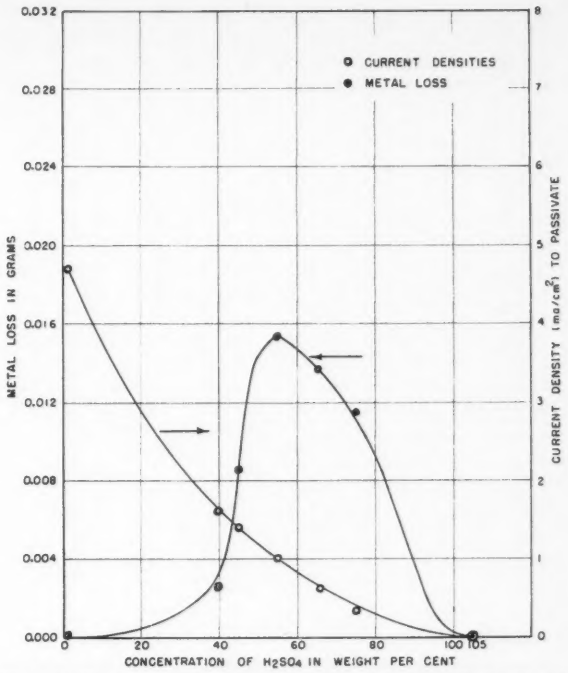

Figure 9—Effect of sulfuric acid concentrations on current density (ma/sq cm) and metal loss of 316 stainless steel at 75 F.

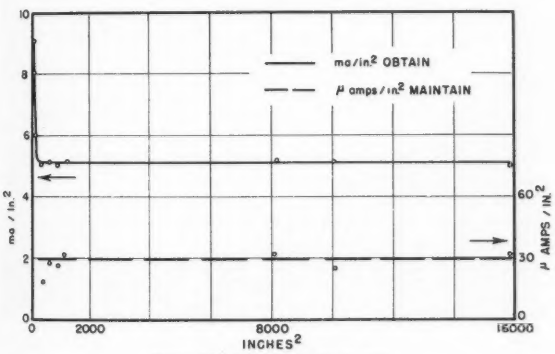

Figure 10—Current density vs area.

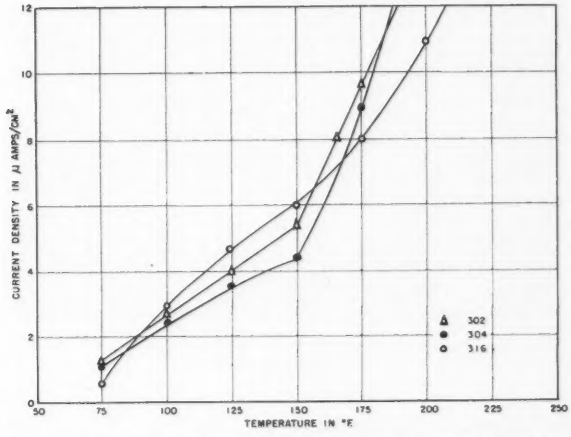

Figure 11—Effect of temperature on current densities of passivated stainless steels in 67 percent by weight sulfuric acid.

certain amounts of these as additives could be tolerated in oxidizing systems. This point was checked with 316 stainless steel in 67 percent sulfuric acid. Varying concentrations up to 2500 ppm of each of the above were added and the system was successfully passivated. The only effect was an increase in current density required to establish and maintain protection. For example, the addition of 2500 ppm $FeCl_2$ resulted in an increase of current to establish passivity by a factor of five and an increase in current to maintain passivity by a factor of 250.

Effect of Agitation. Several qualitative experiments were made to show that agitation would not adversely affect the ability to passivate a system. In fact, the current density requirements may even decrease. In some systems, there seems to be a definite improvement in the rate at which the passive barrier is formed in a large agitated vessel.

Throwing Power

If anodic protection is to be of any practical value it is necessary that protection be possible in the complex geometry of process in vessels and flow lines. All of the preliminary experiments indicated that the "throwing power" of the electrochemical cell involved is extremely good. This seems reasonable since the resistance of the anodic film was shown to be extremely high. An experiment was designed to give a more definite answer to this conclusion. The experimental set up is shown in Figure 12.

A 60 ft. piece of ¾ inch 304 stainless steel tubing had thirteen 180 degree bends and two 90 degree bends. A centrifugal pump and a 20 liter reservoir of sulfuric acid were used to circulate acid through the piping. Acid was circulated for 17 hours with no protection. Periodic samples were obtained and analyzed for iron content. The rate of iron pick up is shown in Figure 13. The iron content increased to over 1000 ppm in 17 hours. The acid was removed and the entire system cleaned by a water rinse. A fresh batch of sulfuric acid was then placed in the experimental system.

In the anodic protection experiments the platinum and calomel reference electrodes were placed at three different locations. In location No. 1 both electrodes

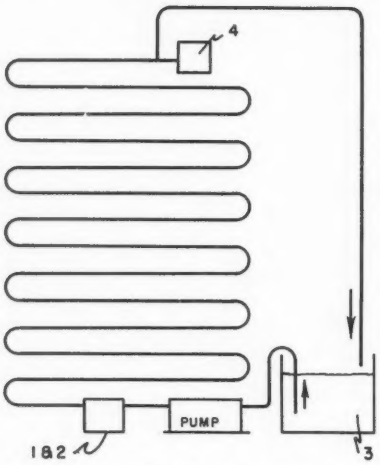

Figure 12—Schematic of throwing-power experiment.

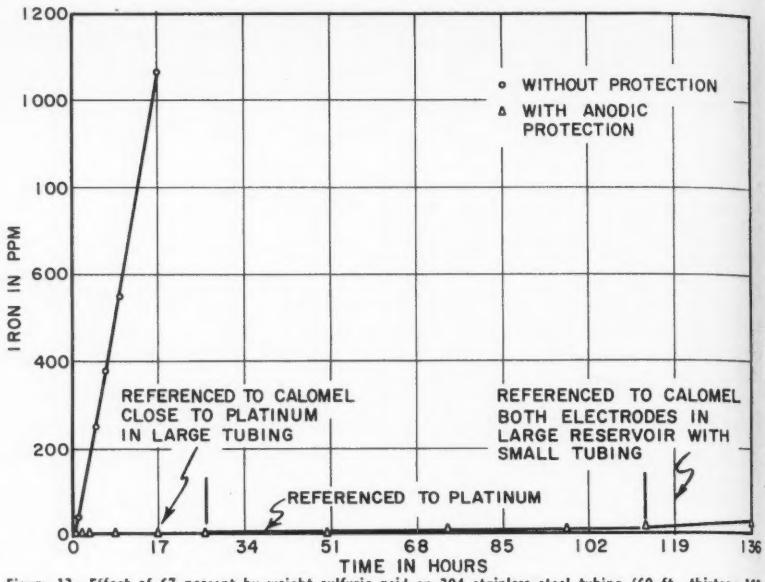

Figure 13—Effect of 67 percent by weight sulfuric acid on 304 stainless steel tubing (60 ft—thirteen 180 degree turns) with and without anodic protection, 75 degree temperature.

Figure 14—Type 310 stainless steel, electrolytically polished and not passivated. Electron diffraction. Approx. 2X.

Figure 15—Type 310 stainless steel, electrolytically polished and passivated. Electron diffraction. Approx. 2X.

were installed in an electrically insulated area remote to the circulating system. No measurable iron was found after 24 hours.

In the second location, the calomel cell was removed and the platinum electrode in location No. 1 was used as both cathode and reference electrode. A very slight increase in iron content occurred in 90 hours.

In the third location, both platinum and calomel cells were installed in the glass reservoir remote to the circulating system. After 24 hours some iron was found in solution but the corrosion rate is still negligible compared to the blank.

At the conclusion of the experiment the stainless tubing was split and the interior examined for any evidence of corrosion on the base metal or at any of the numerous welds in the system. No evidence of damage was seen.

In this very difficult system it must be concluded that anodic protection is both possible and a very practical thing. During this experiment, potentials were measured at various locations along the tubing. When the potential was 0.8 volt near the electrode system the potential was 0.4 volt at the most distant points possible from the electrodes. Since both of these potentials are in the passive range as defined by Figure 1, the entire system remains passive. This conclusion has been substantiated by subsequent field work.

Identification of Protective Film

The properties of the film formed in passivation were examined using the electron miscroscope. The particular advantage of electron diffraction was the very shallow penetrating power of the electron beam. This results in diffraction of electrons by a relatively few atomic layers at the metal surface. The depth of the electron beam has been estimated to range between 10 and 20 Å.[27]

Thus, films of only 10 Å thickness could diffract enough electrons to give a useful pattern on a photographic plate. Amorphous film could not be identified by this method.

Of the stainless steels examined most of the electron work was confined to the austenitic group (Type 310 stainless steel). More emphasis was placed on 310 because its diffraction pattern is more distinct and makes possible a more positive identification of any passive layers. Using a steel specimen prior to passivation most of the lines of Fe-Ni-chromite appear along with the distinct pattern of austentite. This pattern is illustrated in Figure 14. After passivation the steel surface does not diffract (Figure 15). No diffraction lines are observed indicating that the surface of the steel has

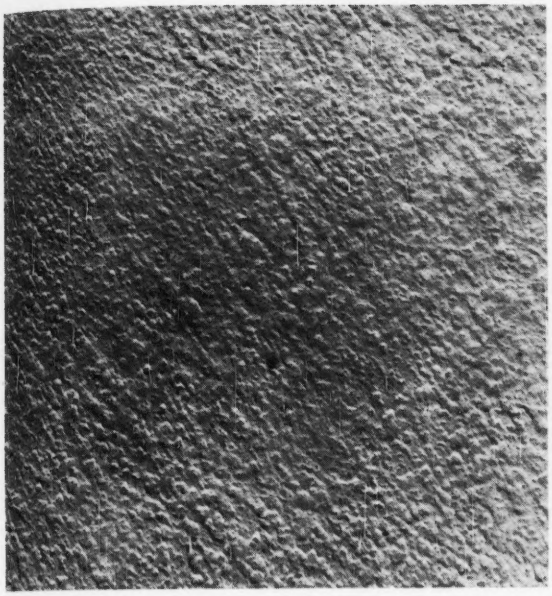

Figure 16—Type 310 stainless steel, not passivated. Electron micrographs, approximately 22,000X.

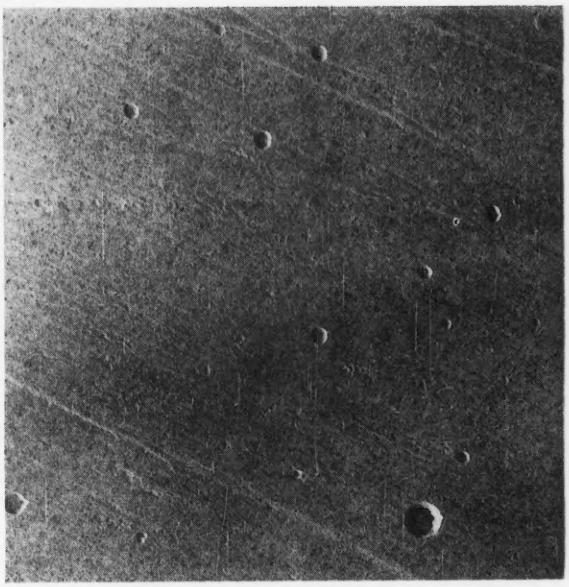

Figure 17—Type 310 stainless steel, polished with Al_2O_3 on wheel passivated 18 hours. Note: fine white lines are associated with excessive amount of chromium used for shadowing. Electron micrographs, approximately 22,000X.

been disrupted by formation of an amorphous layer.

Electron micrographs of these surfaces were determined. A 1½ percent parlodion was used to form a transparent replica of the surface. Micrographs were then made using chromium shadowing techniques. Photographs taken before and after passivation are shown in Figures 16 and 17. It can be readily seen that change in the appearance in the surface has occurred. It appears that passivity has caused a considerable reduction of surface roughness. This could be due to removal of the high spots or the addition of a smooth film. Since no electron diffraction pattern was obtained it is presumed that the surface was smooth and the amorphous film deposited. Based on this exploratory and incomplete work it seems that the crystalline oxide surface of stainless steel is destroyed and amphorous, inert oxide film is formed. This work is discussed in more detail in a paper recently submitted for publication.[28]

Cracking Induced by Anodic Polarization

Some writers have indicated that, in some systems anodic polarization induces stress corrosion cracking. Hines and Hoar[29] studied the cracking of 18-8 stainless in 42 percent $MgCl_2$ at 150 C. They concluded that anodic polarization with current densities of up to 50 $\mu a/cm^2$ decreased the induction period for formation of cracks. The rate of crack propagation is not increased but the total number of cracks formed did increase. Romanov and Dobrolyubov[30] also studied the effect of anodic and cathodic polarization on the rate of stress corrosion of 18-8 stainless in 42 percent $MgCl_2$. They concluded that anodic currents produced a large number of cracks with sharp peaks and numerous branches. Logan[31] showed an increased intercrystalline corrosion when ship plate was exposed to boiling NH_4NO_3 and 60 $\mu a/cm^2$. All these papers reported decreasing cracking with cathodic currents.

None of these systems are directly applicable to the authors' work. However, they do serve as a warning of the possibility of cracking induced by anodic passivation in some systems. Qualitative examinations for cracking have been made throughout the three years' research in this field. No direct evidence of induced cracking has been noted, although certain observations can be reviewed.

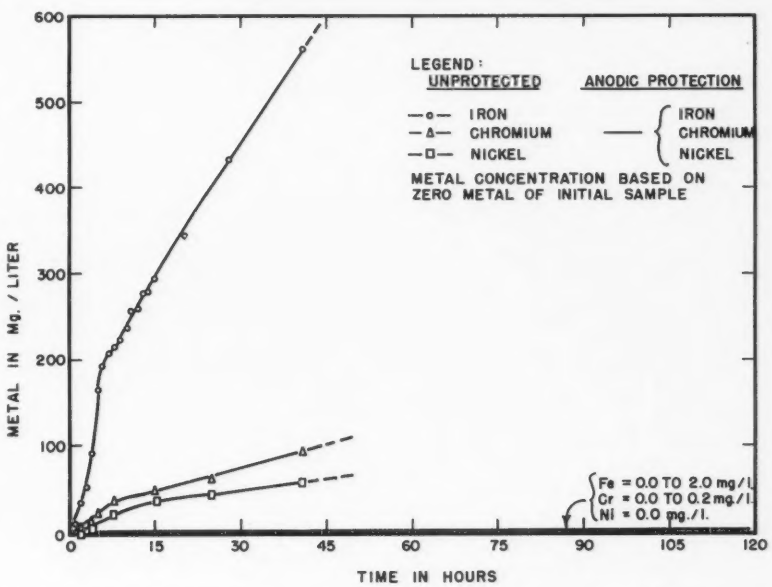

Figure 18—Corrosion of stainless steel tank by sulfuric acid (67 percent by weight) with and without anodic protection.

One 18-8 stainless steel beaker was passivated dozens of times in 67 percent H_2SO_4 during demonstrations. Another was passivated for nine months as a demonstration. Both were cut and examined metallurgically. No evidence of cracking could be found. Further examination was made of the base metal and welds from the ¾-inch tubing used in the throwing power experiment and the 500 gallon milk can. Some hairline cracks were detected at the welds. However, these same type cracks were found on the exterior of the vessel.

Numerous combination stainless weld-

ing rods were used in preparing weld specimens. These were all passivated for several days. No weight loss occurred, nor were any cracks detected.

Based on these data it seemed safe to proceed with anodic protection installations. The authors feel, however, that extensive research in this area must be carried out before any installations are made when high stress levels are likely.

Pilot Test in 500 Gallon 304 Stainless Steel Milk Tank

All of the work reported previously has been based on establishing passivity on relatively small surface areas. In order to evaluate this technique on a large scale a 500 gallon stainless steel tank was selected as a pilot process vessel. A six volt lead storage battery was used as a source of current and the high current capacity controller used to maintain control with the tank filled with 67 percent sulfuric acid. An oscilloscope was used to measure the current required in the experiment. This tank was passivated with no difficulty. The tank had a total surface area of 70 sq ft or approximately 10,000 square inches. The current required to establish passivity was 4.2 amperes applied for about 1/30 of a second duration. The current required to maintain passivity was approximately 142 milliamps, or a current density of about two milliamps per square foot. With time this current requirement declined to a much smaller value.

In order to evaluate the benefit of this technique in actually preventing corrosion, the rate of iron solution was measured with and without anodic protection applied (Figure 18). It is readily seen that there is almost a complete elimination of corrosion in this tank for a period of several days.

Conclusions

1. Passivity induced by anodic polarization was shown to be possible in a wide variety of oxidizing systems.

2. For the first time, anodic passivation has been made practical by the invention and development of an anodic protection system with its high current capacity.

3. This technique seems to be of immediate practical value in stainless and mild steel equipment used to store, process and transport such corrosive chemicals as H_2SO_4, H_3PO_4, NaOH, LiOH, $Al_2(SO_4)_3$ and NH_4NO_3.

4. Laboratory evaluations of any particular system can be extrapolated with reasonable confidence to field performance.

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DISCUSSION

Questions by M. C. Forbes, Houston, Texas:

1. Since the auxiliary electrode is a cathode, why was it necessary to use platinum in commercial installations?

2. The auxiliary electrodes shown were quite small—doesn't this create a problem in concentration polarization due to high cathodic current densities?

Replies by J. D. Sudbury, O. L. Riggs, Jr. and D. A. Shock:

1. We chose platinum because of its inertness and its low resistance value. We chose not to run the chance of metal contamination of product. However, other metals can be used which would be relatively inert to respective systems since they would possibly be cathodically protected. One would have to examine relative inertness of metals to the various systems prior to selection of metal for use as cathode. It is believed, for example, that copper would be useful in H_2SO_4, and nickel would be useful in NaOH.

2. Comparatively speaking the electrodes were small, but we overdesigned the platinum cathode 3X that calculated as necessary. A problem in concentration polarization could very well develop if the location of the reference electrode proved critical. If the reference electrode was located out of the potential range, the instrumentation would seek to anodically polarize beyond its limits (throwing power); consequently, a high current density area would develop immediately surrounding the cathode. This high current area would corrode at an accelerated rate (burn-out). With reasonable care, this problem will not be encountered.

Questions by J. F. Mason, Jr., The International Nickel Co., Inc., New York, N. Y.:

1. What are the effects of turbulence or velocity in offsetting anodic polarization?

2. In super-saturated solutions containing solids such as caustic with entrained salt crystals, what effect would the erosive characteristics have in offsetting anodic polarization?

3. Can you anodically polarize nickel and high nickel alloys?

Replies by J. D. Sudbury, O. L. Riggs, Jr. and D. A. Shock:

1. Our experience supports the assumption that velocity tends to promote passivity when the vessel is under the impressed anodic polarization. In fact the "passive surface substance" tends to have an increased film life when formed in the turbulent system.

2. In our sulfonation plant we have a system under protection which is as you question. The anodic control system has reduced the originally very severe corrosion to a level that iron is not detectable above the background. However, the life of the reference electrode is decreased due to erosive nature of the turbulent solids.

3. Pure nickel and high nickel alloys are not considered to be made passive using the technique of anodic polarization. Erratic unstable potentials have resulted from all our nickel polarization work.

Questions by G. A. Marsh, The Pure Oil Co., Crystal Lake, Illinois:

Can passivity be achieved by initially applying a surge of current to place the specimen into the transpassive range and then gradually backing off the applied current until the potential reaches the passive zone?

Replies by J. D. Sudbury, O. L. Riggs, Jr. and D. A. Shock:

Yes. Decreasing applied current after an initially applied high current (which places specimen in transpassive state) invariably results in promoting a passive potential. However, if sufficient current is not applied initially to cause the potential to shift beyond the "Flade" point, the passive state is never reached. We might also add that the minimum time for reaching passive potential is achieved using the maximum current density. In other words, the energy consumed (coulombs) is directly dependent on time and current/unit area.

Application of Anodic Protection in the Chemical Industry*

By D. A. SHOCK,* O. L. RIGGS* and J. D. SUDBURY*

Abstract

This paper presents the factors which are important to consider in evaluating a given metal-electrolyte system for the application of anodic protection. The general requirements are discussed first, and then a number of examples are given. The anodic protection process is applicable to highly conductive solutions which exhibit the property of establishing a passive range with the metal in question. In general, the oxy acids, the bases and the salts are applicable. The greatest class exclusion are those solutions containing the halogens. The effects of temperature, type of alloy and economic aspects of the system are discussed. Data is presented to show that used with the 18/8 stainless steels the anodic protection process could be applied to HNO_3, H_3PO_4, NaOH, $Al_2(SO_4)_2$ and NH_4NO_3 solutions. 5.9.4

Introduction

THE PHENOMENON of anodic protection has been regarded until now as little more than an interesting laboratory curiosity. Its lack of ready acceptance in industry stems from the special conditions necessary for its application and the dangers inherent in its misuse. If improperly regulated, an anodic protection system may actually increase rather than decrease the corrosion rate.

One industrial application of anodic protection has been reported.[1] It involves the successful control of corrosion in pulp and paper digesters by anodic currents during a pilot test. In this instance the potentials were not controlled by automatic instrumentation.

Design Requirements

In designing an anodic protection system, there are three critical points: (1) the initial current requirement or the height of the Flade potential, (2) the limit of the passive range which controls the design of the voltage control, and (3) the current required for maintenance of the passive film.

At the moment, the anodic protection system requires approximately a 50 millivolt passive range in order to maintain the passive film. This means that one must have a flat portion of the polarization curve** for a minimum of 50 millivolts. The total maximum current has not been ascertained; however, up to 300 amps have been used in an installation for a short time.

Maximum Current Requirements

The absolute maximum initial current required to bring to the passive state has been found to be difficult to determine in many systems because of the very short time required to go across the Flade region. In some cases this is in order of milli-seconds. There is a time relation between current required and achievement of passivity, whereby a large current is required to passivate a given surface quickly, but smaller currents will passivate if a longer time is allowed.

Figure 1 shows the relationship between time to passivation and current at two different surface areas in the sulfuric acid-stainless steel system. It is obvious that less time is required at the higher applied currents. The amount of current required per unit surface area is constant in a given system. Thus, with the unit area requirements determined by laboratory experiment, design can be made for plant equipment. Experiments thus far have shown that the surface of the metal to be protected does not have to be especially cleaned or treated. Passivity is more slowly established on mill scaled and dirty metal; however, the surface preparation does not seem to be a critical factor.

Passive Range Limitations

At present the system limitations are such that a 50 millivolt passive range is needed. Thus the metal-electrolyte systems, which do not have this long a passive range, are not susceptible to automatic passivation. The range is, of course, indicated by the laboratory determination so that the feasibility of a system can be ascertained in the laboratory.

Another aspect of this limitation is the length of time required to reach a minimum value of a point in the passive range. Figure 2 shows a major time difference observed between passive film behavior in acid and in alkaline solutions. The polarization curves run in acid give minimum readings in short enough times that the data for the whole curve can be run rapidly. Furthermore, the values, once they have reached a minimum, do not change appreciably. The LiOH system, however, approaches a minimum at a much slower rate. Values taken at five minutes would be in considerable error. The assumption that a value taken at a short time can represent the maintenance current required would be in considerable error. The time lag factor was of great importance in the NaOH-H_2SO_4 neutralization system in Baltimore,*** as the initially determined current requirements for NaOH were higher than found necessary in practice.

Film Maintenance Current Requirements

The economic limit of applicability comes largely in the third factor in the

★Submitted for publication October 20, 1959. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Denver, Colorado, October 14, 1959.

* Continental Oil Company, Ponca City, Oklahoma.

** See J. D. Sudbury, Anodic Passivation Studies. *Corrosion*, 16, 47t (1960) Feb.

*** See O. L. Riggs. Anodic Control in a Sulfonation Plant. *Corrosion*, 16, 58t (1960) Feb.

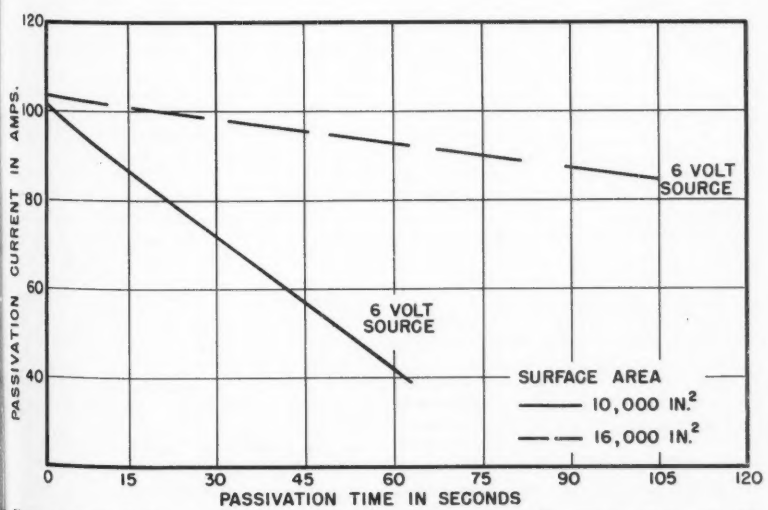

Figure 1—Effect of time on the current required to establish passivity (67 percent sulfuric acid—304 stainless steel).

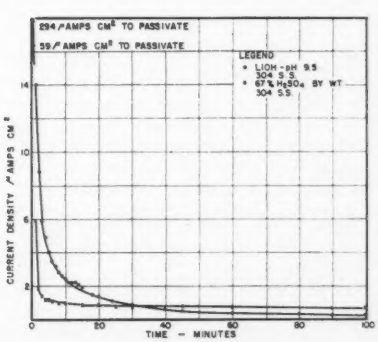

Figure 2—Time of passivation.

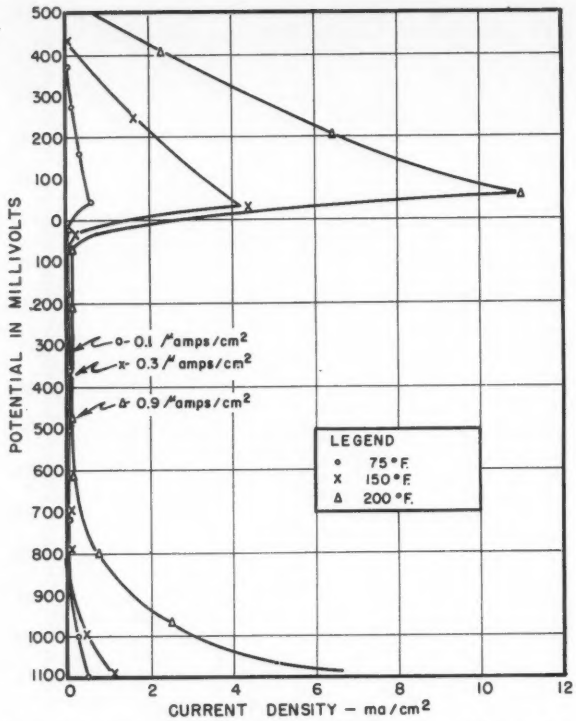

Figure 3—Effect of temperature on anodic polarization (316 stainless steel in 67 percent sulfuric acid). Calomel referennce electrode.

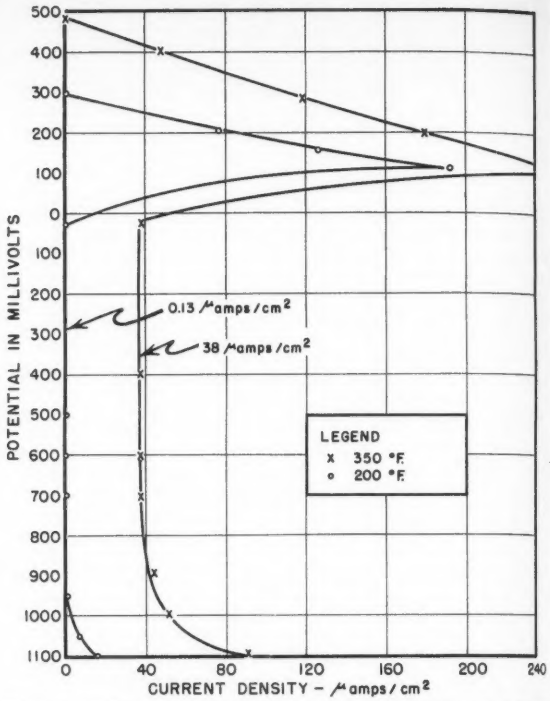

Figure 4—Anodic polarization of stainless steel (316 stainless steel in 115 percent H_3PO_4). Calomel reference electrode.

TABLE 1—Current Density Required to Maintain Passivity of Various Metals and Calculation of Apparent Resistance of Passive Layer (67% H_2SO_4)

ALLOY	Cr/Ni Ratio	Current Density—Microamps Per Cm^2	Total Film Resistance Ohms Per Cm^2
302	(18,8)	2.2	500,000
304	(18+8)	3.8	224,000
310	(24—20)	0.5	2,100,000
316	(18—10+Mo.2)	0.1	17,500,000
405	(12—14)	2.7	333,330
410	(12—.5)	10.9	100,900
446	(25—.5)	0.7	1,786,000
Mild Steel	(SAE 1020)	15.0	26,000
Titanium		0.08	17,500,000
Hastelloy B	(60 + Mo 30)	0.5	180,000
Carpenter 20	(25—20) (Mo 2.5 Cu 3.5)	0.03	46,000,000

TABLE 2—Current Density Requirements for 304 Stainless Steel in Different Chemical Environments

SYSTEM	Current Density μ Amps/In² To Obtain	To Maintain
LiOH [pH of 9.5]	516	0.14
NaOH [20% Caustic]	30,000	64
H_2O_2	64	1.3
NH_4NO_3	580	5.1
$Al_2[SO_4]_3$	580	5.2
H_2SO_4 67% 75° F	3300	0.6
180° F	30	1.9
H_3PO_4 115% 75° F	0.1	0.001
180° F	0.2	0.001
350° F	416	14.0
HNO_3 80% 75° F	16	0.2
180° F	75	0.7

current requirements to maintain passivity. This minimum value results from a combination of factors such as the metal used, the properties of the electrolyte, and, as cited before, the temperature.

Table 1 shows the difference in current requirements in maintaining the passive film on various alloys in 67 percent H_2SO_4. It is noteworthy that the 316 stainless is considerably more easily passivated and produces a much more resistant film than do the other equivalent stainless series. The data also show that, if one is willing to accept the cost of current and increased size of passivating equipment, mild steel might be used instead of stainless. By taking the current density value and potential value between anode and cathode in the passive region, the apparent resistance of the film region, the apparent resistance of the film in ohm/cm^2 is calculated. These values are given in the last column in Table 1 for the various alloys. The resistance of this film has significance in relating the distance one can separate the electrodes. This is important in attaining protection in a complex piece of equipment. With the throwing power resulting from the passive layer on 304 stainless in 67 percent H_2SO_4, it was possible to protect 60 feet of ¾ inch stainless tubing through bends. Protection was first established with electrodes in the reaction vessel at location 1 and 2. Then cathode was moved to 4 and finally location 3 where the entire system was passivated from a remote location.

Effect of Temperature

The temperature of the system places limitations on the anodic passivation method by changing all three of the critical design points. Figure 3 shows the anodic passivation curves of 316 stainless steel in 67 percent H_2SO_4 at three different temperatures as an illustration of temperature effects. It will be noted that an increase in temperature increases the initial passivation current, increases the maintenance current, and shortens the effective passive voltage range.

Summary of Engineering Requirements

In summary, the general method in investigating the possibility of using the anodic protection process is to anodically polarize the proposed metal in the corrosive environment through the temperature range in which the vessel will operate. From the data obtained and the surface area to be protected, the total current for obtaining and maintaining passivation can be determined. If the initial passivating current re-

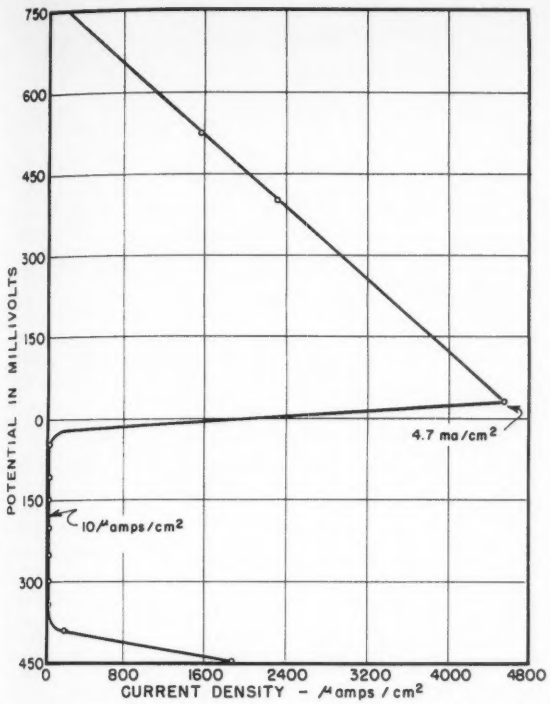

Figure 5—Anodic polarization of stainless steel (20 percent NaOH—deionized NaOH—75 F). Calomel reference electrode.

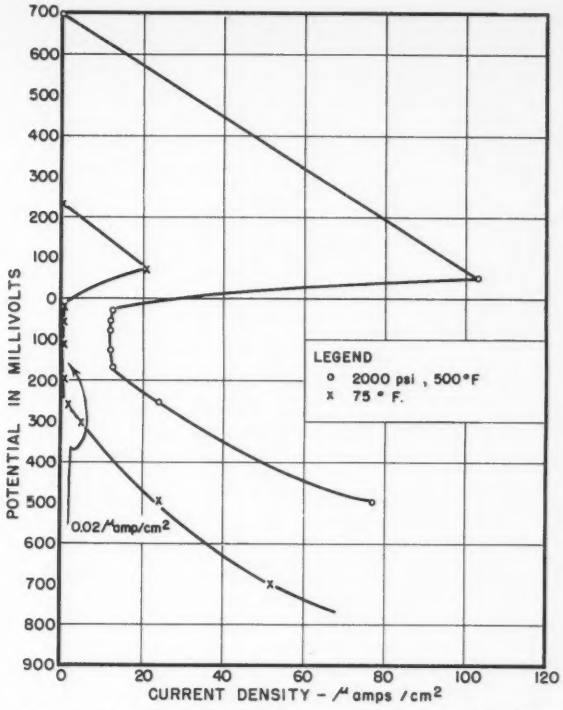

Figure 6—Anodic polarization of 304 stainless steel (LiOH at pH 9.5). Calomel reference electrode.

quired is in the order of several hundred amps and the passive range of not less than 50 millivolts, and maintained with a small minimum current, the anodic protection process can be installed to make and maintain a passive surface. The cost will depend on the current required to maintain passivity and capital cost in the size and complexity of the controller equipment.

Possible Applications

Sulfuric Acid

The case of sulfuric acid has been covered in the previous papers so that it will not be specifically discussed here. It appears that any concentration of sulfuric acid can be kept in any of the 18-8 stainless steels with practically no corrosion using the anodic protection process. As the concentrations of H_2SO_4 get higher (+ 95 percent), carbon steel becomes practical and storage of oleum in carbon steel tanks under anodic passivation gives complete protection and no iron pick-up in the oleum.

Phosphoric Acid

Phosphoric acid is known to be corrosive to the stainless steels. While a wide range of concentrations have not been investigated it appears that anodic protection is feasible. Figure 4 shows the polarization curves at 94 and 176 C of 316 stainless in 115 percent H_3PO_4. From the curve it can be noted that the values to maintain passivity are relatively low, and the voltage range of the passive area is quite large. The current to passivate is not too great in the average size process vessel to require excessive passivation currents. Thus, in this system it is concluded that anodic passivation is feasible.

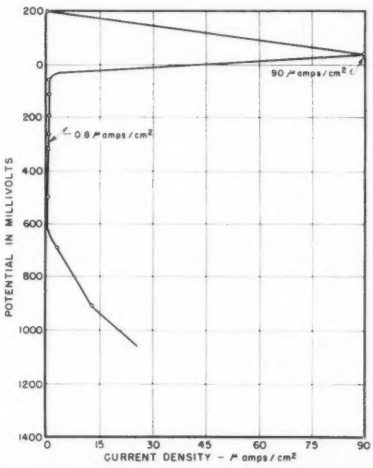

Figure 7—Anodic polarization of 304 stainless steel (deionized water—$Al_2(SO_4)_3$—75 F. Calomel reference electrode.

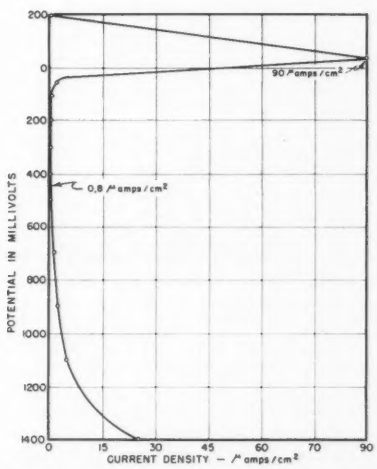

Figure 8—Anodic polarization of 304 stainless steel (deionized water, NH_4NO_3 saturated at 75 F). Calomel reference electrode.

The Alkalies (NaOH, LiOH)

As mentioned, the passive film behavior in the alkaline range is not like the acid ranges. While a thorough study of the passive films in alkalies has not been made, the authors are convinced that the film has certain major differences. As noted previously, there is a longer time lag in establishing the film and minimum passivating current. Figure 5 shows the polarization curve for 304 stainless in 20 percent NaOH. Figure 6 shows the curves for 304 stainless steel on LiOH at 9.5 pH. This system is near the limit of feasibility in that the passive range is not much greater than the minimum 50 millivolt control limit.

Salt Systems $Al_2(SO_4)_3$ and NH_4NO_3

Of the corrosive salt systems which may have some commercial importance aluminum sulfate and ammonium nitrate have been studied (see Figures 7 and 8 with 304 stainless steel). Both of these cases indicate that there should

be no problem using the anodic control process to establish anodic protection.

Nitric Acid

Limited data have been developed to show that nitric acid is very easy to passivate and should present no problem by itself. Considerable effort has been made to passivate the halogen acid-nitric acid systems with meager success. Data indicate that the limit of halogen content in concentrated HNO_3 is about 3 percent HCl, with the 304 stainless steels. At the present state of development, this is not a good system in which to apply anodic protection.

Summary of Applications

Table 2 summarizes the data on a number of systems amenable to anodic protection. This gives some comparative values of currents required to obtain and to maintain passivity. It is significant to note that the system in which the authors made the industrial application (67 percent H_2SO_4—20 percent NaOH) had the highest current requirements. It is felt that in proving the applicability in the sulfuric-caustic system, the passivation has met one of the most rigorous requirements.

Conclusion

It is realized that while considerable work has been done in surveying a number of possible corrosive systems, evaluation has been made of only a few of the possible industrial applications. This anodic protection process should be attractive to those handling H_2SO_4, H_3PO_4, HNO_3, and NaOH, particularly where iron contamination is a problem. In the processing field, the utility of using anodic protection in sulfonation-neutralization equipment has been demonstrated. In handling nitrate and sulfate salts, both (NH_4NO_3, $Al_2(SO_4)_3$), and in some of the fuel element uranium recovery processes one should find definite application for anodic passivation. Mueller described a manual passivation method[1, 2, 3] in alkaline digesters of the paper pulp industries. It is felt that the automatic anodic control system should find application here.

The authors believe that anodic passivation, properly instrumented and engineered, can become another major tool for the corrosion engineer. It is hoped these papers have opened the horizon on a new field of corrosion mitigation.

References

1. W. A. Mueller. *Pulp and Paper Magazine of Canada*, T3-T8 (1959) Jan.
2. W. A. Mueller. *Canadian J. of Tech.*, 34, 162 (1956).
3. W. A. Mueller. *Pulp and Paper Magazine*, T3-T31 (1959).

DISCUSSION

Questions by A. V. Alessandria, Arabian American Oil Co., New York, N. Y.:

1. One of the requirements mentioned for inducing anodic passivation is a liquid to metal contact. Can this system of protection be effectively applied to vapor zones?

2. Can anodic protection be applied to systems made up of various ferrous alloys?

Replies by D. A. Shock, O. L. Riggs and J. D. Sudbury:

1. We have not tried to apply anodic passivation to vapor space corrosion because of the apparent difficulties. If one would contemplate using anodic passivation in vapor space corrosion control it would be necessary to establish a conductive film which is maintained long enough to establish and maintain the passive layer. It would seem to the authors that while this is not impossible, it would be difficult to engineer from a practical standpoint.

2. In order to be positive that the passive range is maintained for all the different types of steel in the system, one should determine the polarization curves for each steel type in the corrosive environment involved. The controls should then be set to operate in the passive range required for all the alloys. In practice, the group of ferrous alloys which are compatible in ordinary metallurgical practice, are usually close enough in their passive range values to warrant no special problems in maintaining proper control.

Any discussion of this article not published above will appear in the December, 1960 issue.

Anodic Control of Corrosion in a Sulfonation Plant*

By OLEN L. RIGGS, JR., MERLE HUTCHISON and N. L. CONGER

Abstract

Using the newly developed automatic potential controller, anodic protection can be used to prevent a severe corrosion problem in the manufacture of petroleum sulfonates. Corrosion was a major problem in a mild steel oleum storage tank and in a 304 stainless steel sulfonic acid neutralization vessel.

Laboratory studies of anodic polarization curves showed that anodic protection was theoretically possible in both these systems. A potential controller, suitable platinum cathode and reference electrode were designed and successfully installed on each vessel. A solid reference electrode was necessary since the usual calomel or copper sulfate reference electrodes were not suited to the system of high temperature and extreme agitation.

The effectiveness of anodic protection was measured in terms of (1) reduction in iron concentration throughout the process; (2) improved quality of product due to absence of colored iron salts; (3) increased productivity of plant due to more rapid product hydrocarbon separation due to the absence of iron soaps. This equipment has continuously and automatically controlled corrosion in a sulfonation plant for one year. 2.4.2

Introduction

INDICATIONS WERE that the anodic protection technique could be used to advantage in protecting units in a sulfonation plant. Before this could be done certain experiments were necessary. Available electrodes were not suited to systems of high temperature and extreme agitation. This made it necessary to design suitable platinum electrodes and develop reliable reference electrodes.

From the results of basic research in the general area of anodic polarization,[1] a new, high-current automatic potential controller was developed. The instrument was designed to fulfill the predicted current demands for the caustic neutralization of sulfonic acids. More particularly it was designed to prevent corrosion of a caustic-sulfonic acid neutralization and the attendant oleum storage tank and blowcase of a sulfonation plant. Major emphasis is placed on the necessary experimental evidence, installation, and evaluation of the controller put to practical service.

Experimental

The neutralization of sulfonic acid with caustic involves a multi-pH system. A metal exposed to this system would then be subject to a range of corroding potentials. Since the anodic polarization technique depends on a range of passive potentials, where little or no corrosion oc-

★ Submitted for publication October 20, 1959. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Denver, Colorado, October 14, 1959.

Hutchison

Conger

MERLE HUTCHISON has been associated with Continental Oil Company since 1948. He has worked in research of core analysis and corrosion involving all phases of petroleum operations.

N. L. CONGER is a member of the Electronics Research Group of Continental Oil Company, Ponca City, Oklahoma. He received a BS in Physics from Tulsa University and an MS in electronics from the Air Force Institute of Technology.

curs, polarization curves should be made for the metal in the acid and the base (Figure 1).

For successful application of anodic protection on a plant, the following conditions are necessary:

1. The range of potentials over which corrosion is extremely low must be reasonably widespread, so as to account for unexpected instrumental fluctuations.

2. The portion of plant to be protected must be in continuous contact with the conductive corrosive fluid (electrolyte) so that current can reach the areas to be protected.

3. It must be possible to maintain all the components of this reaction at some common passive potential.

4. Sufficient power from current supply to over-ride the increased demands of higher temperatures during neutralizations.

5. An accurate, reliable response to reference electrode so that the deviation from the desired potential would be less than 5 mv. (preferably in 0.1 to 0.5 mv range.)

Laboratory

Corrosion Studies. This work stems from the fact that stainless steels although highly resistant to many corrosive systems, are not entirely adequate for all applications. Since the stainless steels do resist corrosion in various environments it is believed tests should be made to determine the relative resistance of a selected group of these metals to H_2SO_4 systems. Time and temperature were used to help express the relative difference in the metals' corrosion resistance. Each metal was prepared for test by shaping to a ½" by 3" coupon. The detection of the severity of corrosion was determined by weight loss and total ppm of iron, chromium and nickel dissolved in the H_2SO_4 solution.

A Similated Neutralization. The vessel used for determining effects of anodic

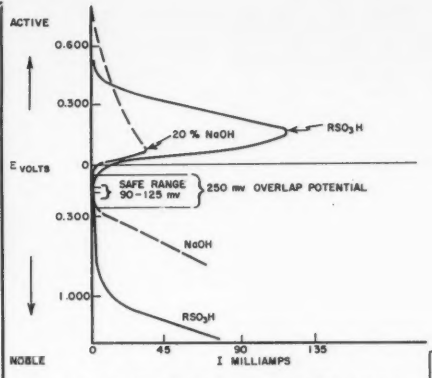

Figure 1—Anodic polarization curves for 304 stainless steel in 20 percent NaOH and RSO_3H.

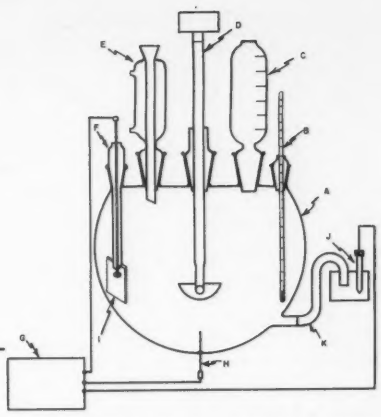

Figure 2—Laboratory RSO_3H neutralization apparatus.

protection during neutralization is shown in Figure 2. It consists primarily of a modified 3 liter, 5 necked, round-bottom flask (A). Suitable openings are required for installing thermometer (B), addition funnel (C), stirrer (D), condenser (E) and specimen mount (F). The potential controller (G) is connected to the various electrodes: platinum cathode (H), metal specimen anode (I) and the reference calomel (J). The electrolytic circuit is completed through an Agar : KCl bridge (K).

The desired amount of 20 percent NaOH was poured into the flask. Enough RSO_3H was weighed into the addition funnel to neutralize the caustic. The funnel was then mounted into the flask. Stirring was begun. At this time the corroding potential was recorded. The potential controller (set to maintain 100 mv potential) was turned on. The evaluation of the experiment was made by: (1) anodic polarization curve from which current density requirements could be

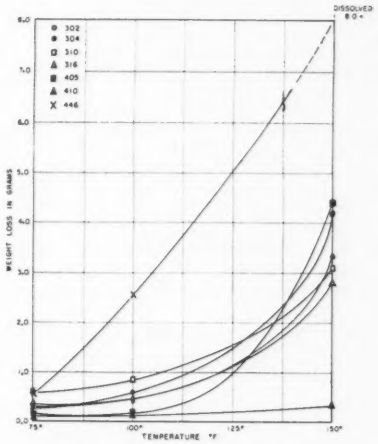

Figure 3—Weight loss of stainless steel in 67 percent by weight sulfuric acid at various temperatures (24 hour exposures).

TABLE 1—Detection of Corrosion Severity

Exposure in Hours	Stainless Steel Alloy Type	Concentration of Metal in P.P.M.		
		Emission Spec.	Corrosion Test Methods	Chemical Analysis
1	302	194	206	210
	304	294	305	303
	316	51	55	54
	410	163	195	195
8	302	461	484	483
	304	426	436	435
	316	455	461	460
	410	318	325	325
24	302	988	1,040	1,040
	304	940	1,013	1,010
	316	1,080	1,100	1,100
	410	422	433	435

TABLE 2—Comparative Observations During Sulfonate Production

OBJECT	Without Anodic Protection	With Anodic Protection
Product	Loaded with a "fluffy" white flocculant matter	Very bright & clear, free of any flocculant matter.
Cuff	Heavy layer of cuff: at least partially due to iron "soap" emulsions.	Greatly decreased initial cuff formation.
Hydrocarbon	Its beginning layer not readily distinguishable. High percentage of both hydrocarbon and product were retained in the cuff.	More distinct sharper breaks in product separation.

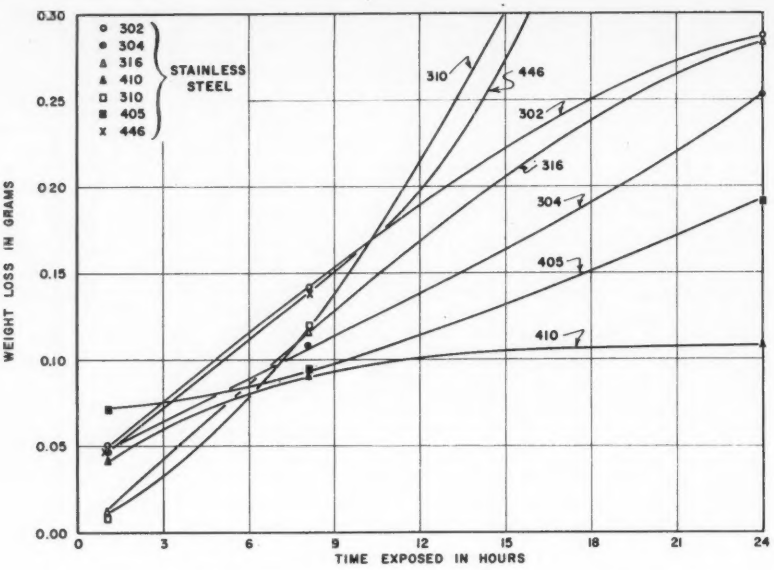

Figure 4—Effect of time on stainless steel exposed to 67 percent by weight sulfuric acid at 75 F.

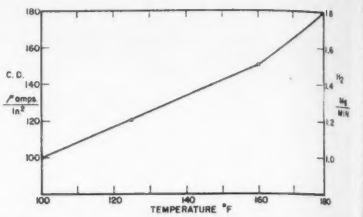

Figure 5—Effect of temperature on current requirements to anodically possivate 304 stainless steel.

markedly affected by time and completely dissolves prior to completion of test at 150 F. These few examples seem to accent the need for critical choice in construction materials of the stainless steels. To add to the dilemma, the type of stainless steel which is most suited to the anodic protection system based on current consumption is Type 316.[2]

After four of the stainless test systems were selected for physical analysis with which to compare chemical analysis, total metal loss was determined. These data are listed in Table 1. The chemical analyses were in very good agreement with the calculations made from weight losses. The emission spectrographic analysis also was in good agreement with weight loss data. It is believed that chemical analysis serves as the best index to corrosion mitigation effectiveness.

Temperature has a pronounced effect on the corrosion resistance of stainless steels. This effect is apparent by the increase in current needs for anodic protection of 304 stainless steel exposed to increasing H_2SO_4 temperatures. Figure 5 shows a gradual increase in current density with increased temperature. The temperature effect is accented at approximately 160 F. It is believed that the passive substances become more affected by solubility properties at that temperature. The passive substances become increasingly soluble with increasing temperatures, calling for more current to maintain them in the passive state.

Costs were calculated for electric current to maintain the Baltimore tank passive. This figure was based on an average current density needed for 304 stainless steel in H_2SO_4 at 75 F. Figure 6 shows how the very small daily (24 hrs.) costs were derived. Even if this calculation were increased 8-fold the cost for electricity would still be a very cheap 6 cents/day.

Figure 7 made use of the mean passive potential (100 mv) obtained from the safe potential range (based on the overlapping passive potential defined by Figure 1) of the acid-base polarization studies. These data represent the current consumed at each interval during the neutralization process in order to passivate and maintain passivity of the 304 stainless steel specimen. The initial current to passivate the metal in 20 percent NaOH was 26 ma (1.5 ma/cm²). The metal was immediately passivated and maintained passive with 300 μ amp/cm². Upon addition of sulfonic acid the current immediately peaked at approximately 1.0 ma/cm² but receded to 30 μ amp/cm² in less than half a second. The current

(1) CURRENT DENSITY 4.3 ma/ft.^{2} (30 μ AMPS./INCH^2)

(2) WATTS = VOLTS · AMPS. [6 · 0.0043 = 0.0258 WATTS/FT.^{2}]

(3) (SURFACE AREA · TANK)(W/FT.^{2}) = TOTAL WATTS
(400 FT.^{2})(0.0258) = 10.32 x 10^{-3} KILOWATT (0.01032)

(4) (Kw)(HRS./DAY) = KwH/DAY
(0.01032)(24) = 0.248 KwH/DAY

(5) (COST/KwH)(KwH/DAY) = (COST/DAY/400FT.^{2} TANK)
($0.03)(0.248) = $ 0.0074/DAY TO MAINTAIN PASSIVITY AT ROOM TEMPERATURE.

Figure 6—Cost to maintain Baltimore tank passive.

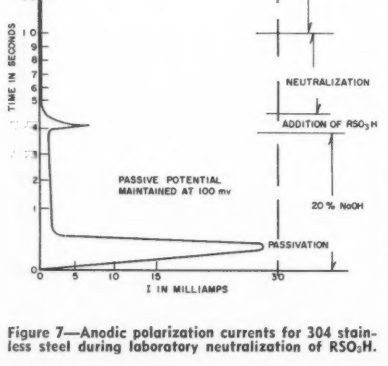

Figure 7—Anodic polarization currents for 304 stainless steel during laboratory neutralization of RSO_3H.

calculated, and (2) change in weight of metal specimen for determining effectiveness of anodic protection.

Detailed cost analysis of consumed current for maintaining the plant passive was needed. This was obtained from pertinent current density measurements. The initial cost, involving instrument and electrodes, were compared to maintenance (repairs and replacement), costs, losses in production, and other less obvious deficits.

Laboratory Results

The effects of increased temperature and duration of metals exposure to the 67 percent H_2SO_4 system are presented in Figures 3 and 4. The inadequacy of randomly selected stainless steel is apparent. One type (i.e., 310) may be much less affected by increased temperature than another type (i.e., 304), with the converse holding true for time. Type 410 is relatively unaffected by time or temperature. Type 446 on the other hand is

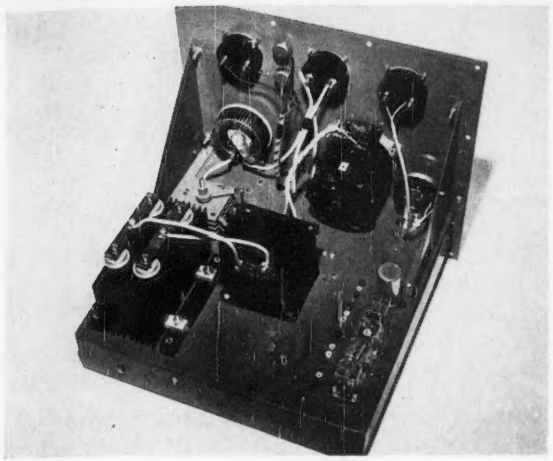

Figure 8—Automatic potential controller (internal view).

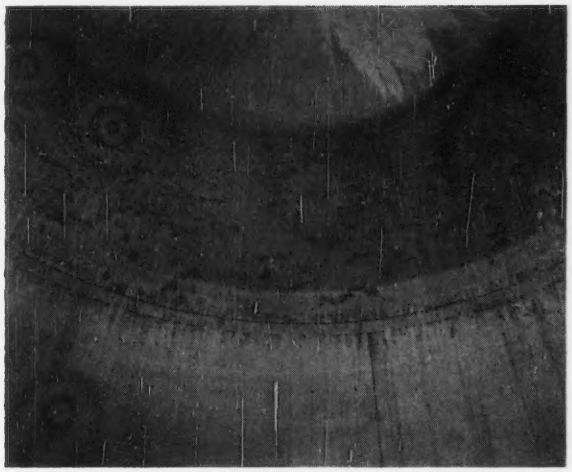

Figure 9—Severe pitting in neutralizer tank.

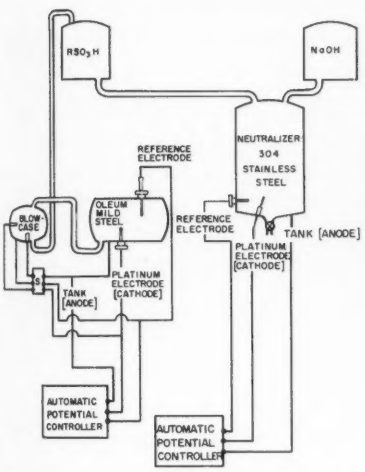

Figure 10—Schematic of anodic protection installation.

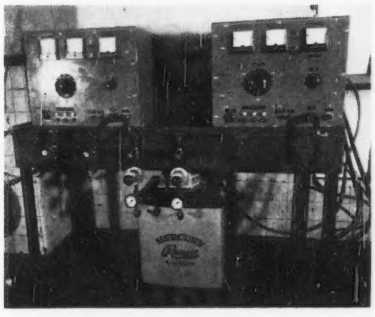

Figure 11—Potential controllers installed.

needed to maintain passivity steadily decreased throughout the neutralization process. These requirements were considered in building controllers (Figure 8) for the sulfonation plant.

The Sulfonation Plant

The units which were to be subjected to anodic protection were the neutralization tank and the oleum storage tank and blowcase. These units were chosen for the test for several reasons. They represented the extremes for systems in which anodic polarization could be put on trial. The oleum is reported to be very mildly-to non-corrosive to mild steel. This is true until condensation of moisture allows a small amount of water to entrain. The anodic protection technique is easily accomplished here. The neutralizer, however, is subject to very severe pitting as shown in Figure 9. The multi-pH system presents a real challenge to the anodic protection technique. Another reason for choosing this system was because it was thought the anodic protection could possibly reduce if not eliminate the operational problem of "cuff formation". This is a very stable emulsion accented by the formation of iron soaps. With the elimination of corrosion, no iron would be available to form the undesired soaps; hence no "cuff formation."

Installation of Controllers

The installation of potential controllers was made according to the schematic drawing in Figure 10. Controllers were attached to the oleum system (storage and measure) and to the neutralizer. The controller on the oleum storage tank would be working at all times until filling of blowcase. The switch between the two tanks would then be engaged to allow protection of the blowcase. The oleum storage tank would not be left without current long enough to lose its passive state. The operation of the controller on the neutralizer would begin as soon as the caustic covered the electrodes. At the end of each neutralization and subsequent removal of products from the tank, the controller would be turned off when the liquid level reached the electrodes. It would then remain off until beginning of next neutralization.

The controllers were ready for service after the electrical cables were mounted (Figure 11), and the electrodes attached to the tank. The relative positioning of the electrodes in the neutralizer tank is shown in Figure 12.

TABLE 3—Iron Analysis of Yield in Sulfonation Plant

Sample		Total Iron in PPM	
Date	Batch	In Charge	In Yield
10/10/57	X-216-217	30	230
11/ 1/57	X-218-219	24	240
2/ 3/58	T-192-193	55	310
After Anodic Protection			
5/20/59	X-155-156	20	19
7/23/59	X-210-211	19	16
8/ 3/59	X-230-231	17	20

Results and Discussion

The effectiveness of automatic anodic protection was measured in terms of (1) reduction in iron concentration throughout the process, (2) improved quality of product due to more rapid product-hydrocarbon separation, (3) reduction in "cuff-formation" due to the decrease in iron soaps which help form the tight emulsion and (4) increase in productivity of plant. The relative descriptions before and after anodic protection are listed in Table 2.

Oleum Storage Tank

The iron concentration had been as high as 1273 ppm. The average iron during normal "10 day on—4 day off" operation was approximately 500-600 ppm. At the time of installation of the potential controller, the concentration of iron was approximately 600 ppm, with tank nearly empty. Upon filling the tank, iron analysis was 450 ppm. The oleum as received contains an average of 30 to 50 ppm iron. Less than a week after anodic protection was started the iron concentration had been reduced to the minimum (39-41 ppm iron). Figure 13 gives the iron analysis of oleum storage tank before anodic protection and for one year following. The iron concentration has remained at the 39 to 41 ppm range continuously.

Blowcase Tank

The oleum samples from the blowcase contained 550 ppm dissolved iron prior to anodic protection. After anodic pro-

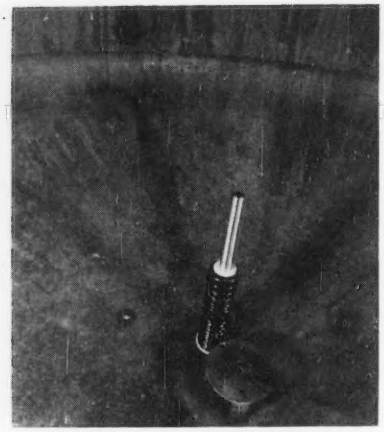

Figure 12—Relative electrode position in neutralizer.

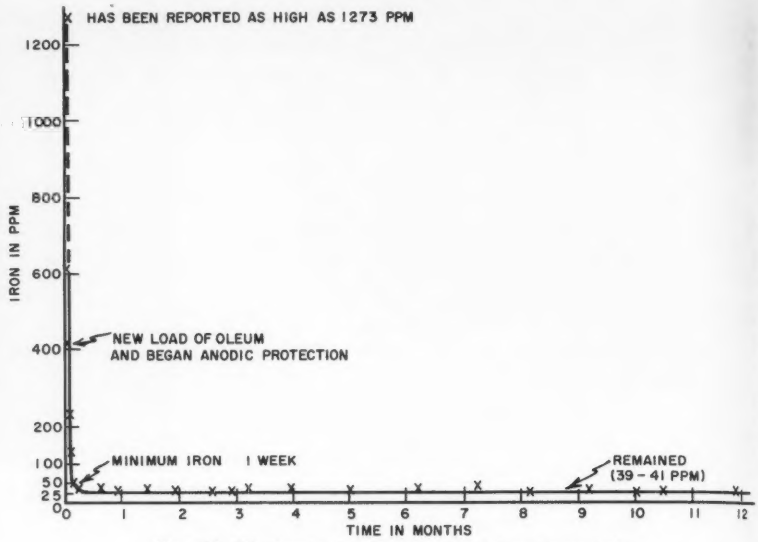

Figure 13—Effectiveness of anodic protection of oleum storage tank.

tection of tank the iron concentration was reduced to 125 ppm in three operation runs. Ten days later the iron concentration was reduced to 59 ppm. To date and following the two week initial treatment period, the iron concentration in the blowcase never exceeded 50 ppm. This tank is under protection only intermittently but the data would indicate adequately protected. It would appear also that one controller can satisfactorily protect two systems through a switching device.

Neutralizer Tank

The multi-pH system of the caustic neutralization of sulfonic acid presents a difficult obstacle for the potential controller. After a final and satisfactory reference electrode was put in service, the controller operated efficiently.

To determine the effectiveness of operation of the potential controller, it was necessary to determine an iron balance for the sulfonation plant. A history of irons was taken in late 1957 and early 1958 (prior to the late 1958 controller installation.) Table 3 shows the great increase in sum total of iron in all phases of yield (hydrocarbon, cuff and product). Also, listed in the table are the iron values for the sum total of all charge ingredients to neutralizer (caustic, oleum, hydrocarbon and water). There is no increase in iron after neutralization with the tank under anodic protection.

Conclusions

1. The anodic protection system has been successfully applied to a sulfonation plant.

2. The corrosion of 304 stainless steel tank used for NaOH neutralization of sulfonic acids can be reduced.

3. The mild steel tank, storage for oleum, and oleum blowcase can be passivated and maintained in non-corroding state with the anodic protection technique.

4. The removal of dissolved iron, the reduction in "cuff formation" and elimination of flock results in much quicker and sharper breaks between hydrocarbon and product layers, giving a higher quality product and a reduced processing cost.

Acknowledgment

The assistance of W. O. Miller, Bill Boone, and Bob Maples, Petrochemical Department, Continental Oil Company, is gratefully acknowledged.

References

1. J. D. Sudbury, O. L. Riggs, Jr., and D. A. Shock. Anodic Passivation Studies. *Corrosion*, 16, 47t (1960) Feb.

Any discussion of this article not published above will appear in the December, 1960 issue.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

A Proposed Standardized Static Laboratory Screening Test for Materials to Be Used as Inhibitors In Sour Oil and Gas Wells

A Report of NACE Technical Unit Committee T-1K*
On Inhibitors for Oil and Gas Wells, Prepared by
Task Group T-1K-1** on Sour Crude Inhibitor Evaluation

NACE TECHNICAL COMMITTEE REPORT

Publication 60–2

Abstract

A description is given of a static laboratory test designed to provide a simple, easily reproducible method for screening materials to be evaluated subsequently as possible inhibitors in sour oil and gas wells. Mild cold rolled steel specimens were immersed for seven days in deaerated brine containing 400-600 ppm hydrogen sulfide plus kerosene. Materials suppressing 90 percent or more of the weight loss found in the control were deemed to be suitable for further study.
2.3.4

1. Purpose of Test

THE PURPOSE of this laboratory test is to provide a simple, easily reproducible method for screening materials for subsequent evaluation as corrosion inhibitors in sour oil and gas wells. A material may pass, fail, or pass conditionally.[1] A material which passes is considered suitable for evaluation, one which fails is considered unsuitable for evaluation, and one which passes conditionally may be evaluated at the operator's discretion.

2. Description of Test

This is a static weight loss test, wherein a weighed coupon is immersed in a prescribed manner in a standard corrodent for one week. This coupon is the "blank" or "control." In an identical system a definite concentration of the material to be tested is added, and this test is run simultaneously with the control. At the end of the one week test period the amount of weight loss suppression afforded by the material added is determined.

3. Criterion for Passing Test

If in the test the material suppresses 90 percent or more of the weight loss found in the control, the material is said to have passed the test. If it suppresses 80 percent but less than 90 percent of the weight loss, it is said to have passed conditionally. If it suppresses less than 80 percent of the weight loss, it is said to have failed.

4. Equipment

(a) Test Container—a 500 ml narrow mouth Erlenmeyer flask (Corning No. 4980 or equivalent).

(b) Closure—a No. 7 rubber stopper. In the center of the bottom of this stopper a glass hook is inserted. This hook is made of 3 mm glass rod, about 5½ inches long, the hook having a radius of about ¼ inch. This hook supports the coupon in the corrosive medium.

5. Corrosive Medium

(a) Brine

Use a 5 percent solution of CP or reagent grade sodium chloride (by weight) in distilled water. The brine is deaerated by introducing pre-purified nitrogen through a sintered glass dispersion tube. Deaeration is usually complete in two to three hours. Hydrogen sulfide gas is then dispersed in the brine until a concentration of 400 to 600 ppm (as determined by standard methods) is obtained.[2]

(b) Hydrocarbon

Use a good grade of kerosene which has been filtered through Fuller's Earth to remove polar materials.[3]

(c) Amount of Fluids Used

Each test or control requires 450 ml of brine and 50 ml of hydrocarbon, a total of 500 ml of fluid.

6. Test Specimen

(a) Description

Mild cold rolled steel, 1/16 inch thick, should be sheared into one inch squares. A hole 5/32 inch in diameter should be drilled about ⅛ inch from the center of one edge of the coupon. Any edge splinters or roughness should be removed by grinding.

(b) Surface Preparation

(1) Use only those coupons which are free from pits and deep scratches.

(2) Using a 240 grit emery cloth belt on a mechanical sander, abrade coupon on both faces until surface appears uniform and no original surface defects or scratches remain.

(3) Brush to remove grit.

(4) Immerse in acetone to remove grease.

(5) Place in desiccator until ready to use.

(c) Weighing

Weigh coupons on an analytical balance to the nearest 0.1 milligram and record weight.

7. Number of Controls and Tests

(a) Tests and controls should be run in duplicate. If several materials are tested simultaneously one set of controls is sufficient.

(b) If weight losses of duplicate tests or controls do not agree within 1.0 milligram or within 10 percent of the greater weight loss, additional tests or controls should be run until such agreement is obtained.

8. Concentration of Material to be Treated

The policy of the test is to compare

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** R. D. Stanphill, Humble Oil and Refining Co., Houston, Texas, vice-chairman.

[1] This test may be adapted to work backward to find a suitable concentration for experimental materials. Thus, if 0.023 gr of material provides 90 percent protection, a workable concentration for field use would be a 46 percent concentration of the material in a suitable solvent.

[2] If the brine clouds upon addition of the hydrogen sulfide, it is an indication of incomplete deaeration, and the entire batch should be discarded.

[3] This may be done by adding about one quart of Fuller's Earth to five gallons of the kerosene in a five gallon container. The mixture should be agitated by rolling, by mechanical stirring, or other means. After allowing the Fuller's Earth to settle, the supernatant clear kerosene can be siphoned off into another container.

materials at equivalent concentrations related to cost. It is based on 100 parts per million of material (related to the total fluid phase—500 ml) at $2.00 per gallon. The test concentration for materials costing more or less than $2.00 per gallon is obtained as follows:

$$\text{Test concentration, ppm} = \frac{200}{\text{Cost of material, dollars/gallon}}$$

If the cost of the material is not known, it should be estimated as closely as possible. (See footnote No. 1.)

9. Procedure

(a) Arrange on a work bench the proper number of flasks (two for each material to be tested plus two controls) and stopper each with a No. 7 rubber stopper in which a glass hook is inserted.

(b) Flush air out of each flask by introducing pre-purified nitrogen through a delivery tube leading to bottom of flask.[4]

(c) Introduce the proper amount of inhibitor directly into flask.[5]

(d) Introduce 50 ml of prepared kerosene into each flask through a delivery tube.

(e) Introduce 450 ml of prepared brine into each flask through a delivery tube underneath the kerosene. This addition should be made slowly to avoid splashing and mixing of the two phases.

(f) Lift the No. 7 stopper, hang a prepared and weighed coupon on the glass hook, support coupon in kerosene layer for ten seconds, then lower into brine layer.

(g) Allow tests and controls to remain undisturbed for seven days.

(h) Remove coupon, dip in acetone, clean electrolytically or in inhibited acid until free of corrosion products, dip in acetone and place in a desiccator until ready to weigh.

10. Weighing

Weigh coupons to nearest 0.1 mg and record.

11. Calculations

Let C = Average weight loss of control coupons.

Let T = Average weight loss of test coupons for each material.

$$\% \text{ Inhibition} = 100 - \frac{100T}{C}$$

(4) It is convenient, especially for large numbers of tests, to utilize a glass tube inserted through the No. 7 rubber stopper to the bottom of the flask for flushing with nitrogen, and for the addition of the kerosene and brine (steps *d* and *e*). The glass tube can then be closed off with a short length of rubber tube and a tubing clamp for the duration of the test.

(5) Any method which allows the addition of the proper weight of material without introducing foreign solvents, coupling agents, etc. may be used for this step. If the material is soluble in kerosene, it may be made up into a 5 percent or 10 percent solution and a known volume pipetted into the flask. A Luer Tuberculin Syringe (1 ml graduated in 0.01 ml), also may be used for materials which are not too viscous. Solid or highly viscous materials may be weighed out on a glass microscope slide cover and added to the flask.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Fourth Interim Report

Use of High Silicon Cast Iron for Anodes

A Report of Technical Unit Committee T-2B*, Prepared by NACE Task Group T-2B-4 On High Silicon Cast Iron Anodes

NACE TECHNICAL COMMITTEE REPORT

Publication 60—3

Abstract

Data are tabulated from questionnaire replies received from more than 100 companies pertaining to use of high silicon cast iron for anodes. Of the 230 installations for which data were reported, 53 percent concerned ground bed applications, 37 percent fresh water and 10 percent salt water.

In general, data indicate high silicon cast iron anodes are at least equal to graphite anodes in ground bed applications where coke breeze backfill is utilized and superior in marshland and other areas where backfill is impractical. In fresh water applications high silicon cast iron anodes perform very well so long as the temperature does not exceed 125 F. At high temperatures the molybdenum containing alloy is preferred as is also the case in many brackish or salt water applications where the temperature may exceed 100 F. 6.2.2

Introduction

THE ANNUAL survey, started in 1955, in which questionnaires were sent to companies using high silicon cast iron anodes for impressent current cathodic protection applications was continued in 1958. For the present survey approximately 200 letters were mailed and replies were received from slightly over 50 percent of the companies contacted. These replies contained information pertaining to more than 230 installations. Of these installations, approximately 53 percent concerned ground bed applications, 37 percent fresh water and 10 percent salt water. Some of the first high silicon iron installations have now been in service five years. Indications are that they will see many more years of satisfactory service.

In general, data indicate high silicon cast iron anodes are at least equal to graphite anodes in ground bed applications where coke breeze backfill is utilized. Where backfill is impractical or other materials have failed because of existing conditions (acidic and alkaline soils, inadequate backfill, etc.) the high silicon cast iron continues to perform very satisfactorily.

The general trend in fresh water applications indicates that these anodes perform very well so long as the temperature does not exceed approximately 125 F. At higher temperatures the molybdenum containing alloy is preferred. The latter material also is preferred for many brackish or salt water applications where the temperature may exceed 100 F. Chloride environments tend to attack the standard high silicon iron anode at an unpredictable and excessive rate when chlorine cannot readily permeate from the anode surface. The molybdenum-bearing variety is much more resistant and predictable though it is not free from attack in all such environments. However, it is showing a definite superiority in applications where the molybdenum free anode previously failed.

As in previous survey reports the installations will be subdivided for simplification into ground bed, fresh water and salt water categories.

Ground Bed

Many ground bed installations (Table 1) utilizing high silicon iron anodes have been in service approximately four years giving very satisfactory service with indications of many more years of satisfactory service to come. This includes installations both with and without backfill, anodes installed in marshland where backfill is impractical, and where a varying water table exists. This trend was recorded in previous reports on high silicon iron anodes and this year's replies further substantiate the trend.

In the early stages of the investigational work on high silicon iron anodes by this committee, the short term test data cited merely provided an indication of the performance of the anodes. However, the fact that many ground bed installations have now been in service over four years with little or no change in operating characteristics tends to establish a pattern from which predictions can be made. Based on this information it appears that high silicon iron anodes can be considered at least equal to other anodes studied for normal ground bed installations. They appear to be superior in severe applications where the other materials have limitations such as in marshland and river crossings where backfill is impractical or where there is a varying water table.

Typical ground bed installations utilizing coke breeze backfill are operating very effectively with little or no change taking place. There are many installations in this category and from all the data provided thus far it appears fairly well established that these installations will continue in a like manner for many years. The life of these coke breeze backfilled installations seems to be extremely long based on service data reported.

In an environment where backfill cannot be used or is impractical to use, high silicon iron can be employed to advantage. The anodes can be pushed into a swampy area or placed on a river bottom (Test 22 and 18 respectively) and provide the cathodic protection that is desired. In both cases it was impossible to use backfill, yet there has been relatively no change in the output with time. It is installations such as these where the high silicon iron anodes show a pronounced superiority.

Inspecting a ground bed installation is costly when the anodes are actually removed from the ground. Consequently, relatively few actual inspections of the anodes have been made. In those instances where they were made, the condition of the anodes was very good except for one case involving a chlorine environment (Test No. 25). Failure of the polyvinyl chloride (PVC) jacket on the lead wire actually caused the bed to fail after approximately three years. However, selective attack at the connection end would have limited the service life obtained from these anodes. Since the molybdenum containing high silicon iron has superior resistance to chlorine attack, replacement was made using this alloy with high density polyethylene insulated cable. The insulation was applied to the lead wire in a single extrusion to the same outside diameter as the formerly used polyethylene-PVC combination. Thus a comparison will be obtained between the two high silicon iron materials and also between the PVC and high density polyethylene.

Test No. 2 is an installation in which the tops of two anodes were uncovered and examined and there was no apparent

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TABLE 1—Typical Ground Bed Installations

Test No.	Anode Material	Anode Size	Anode Position	Backfill	Total Bed Resistance, Ohms	Soil Resistivity, Ohm-Cm	Total Current, Amperes	Average Current Density, Amp/Ft²	Approx. Amp. Hrs. per Anode	Remarks
1	4-HSI	2″x 60″	Vert.	Coke Breeze	1.2		6.0	0.575	46,700	Current discharge is 1.5 amps for each HSI anode and 1.1 amps for each graphite anode. Forty-five mos. service and still operating satisfactorily.
	8-GR	3″x 60″	Vert.	Coke Breeze			9.0	.282		
2	15-HSI	2″x 60″	Vert.	Coke Breeze	0.116	350	68	1.75	165,000	The tops of two anodes showed no attack when uncovered after 3½ yrs. Bed in operation 4 yrs. Previous graphite bed failed in 5 yrs.
3	12-HSI	2″x 60″	Vert.	Coke Breeze		500-1000	59	1.9	60,000	Sixteen mos. acceptable operation in alkaline soil. Calcium chloride then used to counteract dry bed and failure occurred within 13 mos. Bed removed from operation.
4	1-HSI	1½″x 60″	Vert.	Coke Breeze	3.2	1240	2.6	1.3	91,100	Coal storage results in acid condition (pH 3.2). Output for first anode constant for 4 yrs. Output of previously used graphite anodes decreased 90% in like period. Anode at lower current density lost in a construction project but was still operable after almost 3 yrs.
	1-HSI	1½″x 60″	Vert.	Coke Breeze		2650	1.2	0.7	29,200	
5	12-HSI	1½″x 60″	Vert.	Coke Breeze	0.8	1000	15.1	0.63	36,000	Forty-four mos. protecting bare line. Good service still realized.
6	3-HSI	1½″x 60″	Vert.	Coke Breeze	21.0	5000	0.7-1.2	0.10-0.20	8,000	Forty-four mos. protecting coated gas main. No change in operating characteristics.
7	54-HSI	2″x 60″	Vert.	Coke Breeze	0.17	2000-10000	83	0.60	27,700	Twenty-six mos. protecting bare line and lead sheathed cable. Installation operating as designed.
8	12-HSI	2″x 60″	Vert.	Coke Breeze	0.24	900	25	0.61	64,300	Over 4 yrs. service with no change. Output reported equal to graphite.
9	2-HSI	1″x 60″	Vert.	Coke Breeze		300	0.1-9.3	0-3.3		Anodes provided identical output for over 3 yrs., but HSI showed decrease in output during 4th year. Anodes to be inspected at earliest opportunity.
	2-GR	3″x 60″	Vert.	Coke Breeze		300	2.5-9.3	0.3-1.1		
10	2-HSI	2″x 60″	Vert.	Coke Breeze		350	1.3-6.4	0.3-1.2		Anodes providing identical service for over 3 yrs. although some decrease in output of HSI anodes has been noticed during fourth year. Anodes to be inspected.
	2-GR	3″x 60″	Vert.	Coke Breeze		350	1.6-6.4	0.2-0.8		
11	10-HSI	2″x 60″	Vert.	None (marsh)	0.31	400	10-14	0.38-0.55		Anodes pushed into swamp. Output difference between HSI and GR anodes almost in direct relationship to area. Total current output for GR decreased slightly from previous report. Anodes in service 3 years.
	5-GR	3″x 60″	Vert.	None (marsh)		400	6.5-7.5	0.33-0.38		
12	1-HSI	1½″x120″	Vert.	Soil		13500	0.80	0.20		Test anodes in service 3 yrs. Graphite and HSI have closely similar outputs in coke breeze when corrected to common conditions. Last report after 2 years operation.
	1-HSI	1½″x120″	Vert.	Coke Breeze		17522	1.12	0.28		
	1-GR	2″x 60″	Vert.	Coke Breeze		16500	0.81	0.31		
	1-HSI	2″x 60″	Vert.	Coke Breeze		21500	0.67	0.26		
	1-GR	3″x 60″	Vert.	Coke Breeze		2550	1.15	0.28		
	1-HSI	3″x 60″	Vert.	Coke Breeze		19600	1.18	0.30		
	1-HSI	2″x 60″	Vert.	Soil		25233	0.44	0.17		
13	2-HSI	2″x 60″	Hori.	Coke Breeze		40050	.62	0.119		Over 3 yrs. service. Output difference between anodes with coke breeze and soil backfill decreasing slightly. Anodes in coke breeze originally passed 75% of total current but now has decreased to 64%. Total output changed slightly.
	2-HSI	2″x 60″	Hori.	Soil		40050	.35	0.067		
14	6-HSI	1½″x 60″	Hori.	None (marsh)	6.6	3000-7000	9.0	0.75	33,000	No change in output in 44 mos.
15	1-HSI	2″x 60″	Vert.	None	2.67	500	115–150	2.4-3.0	140,000	High current density. Single resistance calculated as $R = 0.005\rho$. Over 2 yrs. successful service.
	19-HSI-Mo	2″x 60″	Vert.	None						
16	9-HSI	2″x 60″	Vert.	Coke Breeze top 3′ of anode	1.07	6000	28	1.2	107,200	Graphite anodes failed after 1½ yrs. No great drop in output with HSI after 4 yrs. Consumption rate 0.89 lbs./amp. yr. Estimated life—10 yrs.
17	102-HSI	2″x 60″	Vert.	Soil	4.0	220-12000	140	0.6	26,400	Four separate beds showed average single anode resistance of about $R = 0.005\rho$
18	16-HSI	2″x 60″	Hori.	None	2.6	2450	8.0	0.19	18,500	Anodes placed in river bottom. Output constant for 42 mos.
19	10-HSI-Mo	2″x 60″	Vert.	None	0.28	550	36	1.4	47,500	Anodes pushed in marshy soil 30 mos. ago. Output unchanged after 18 mos. No report since that time.
20	18-HSI	2″x 60″	Vert.	None	1.20	1300	28	0.55	49,000	Anodes jetted into place and after 4 yrs. are still performing satisfactorily. Bed resistance reduced to about 0.80 ohms.
	2-HSI-Mo	2″x 60″								
21	16-HSI	1½″x 12″	Hori.	None			0.13	0.02		Small duct anodes in series showed no change after 34 mos.
22	4-HSI	2″x 60″	Hori.	None	10.0	2000	6.4	0.62	34,000	Anodes pushed in swampy soil. Installation still satisfactory after 42 mos.

HSI-High silicon iron.
HSI-Mo—High silicon iron containing molybdenum.
GR-Graphite.

TABLE 1—Continued

Test No.	Anode Material	Anode Size	Anode Position	Backfill	Total Bed Resistance, Ohms	Soil Resistivity, Ohm-Cm	Total Current, Amperes	Average Current Density, Amp/Ft²	Approx. Amp. Hrs. per Anode	Remarks
23	50-HSI	2″x 60″	Hori.	None	0.42	50–400	130.0	1.0	60,000	Three beds varying in service from 30 to 42 mos. All operating with no change.
24	44-HSI	1″x 60″	Hori.	None	0.27	570–800	92	1.5	53,500	Two beds placed in a water environment. Service: 33-36 mos.
25	2-HSI	2″x 60″	Hori.	None	1.95	514	13.5	2.6	172,000	Lead wires on anodes failed after about 3 yrs. with consumption rate of 1.12 & 1.17 lbs./amp. yr. respectively. Selective attack at connection end would have caused failure before anodes were consumed. Because of chlorine environment, replacements are 2″x60″ HSI-Mo with high density polyethylene cable.
26	10-HSI	1½″x 60″	Vert. (290–350′ depth)	Coke Breeze	1.0	1500–1800	10	0.5	17,000	In service 2 yrs. with no evidence of gas blocking or increased anode-to-earth resistance. One of seven such installations.
27	1-HSI	2″x 60″	Hori.	None			0.7	0.3	25,000	After 4 yrs. service the GR with no backfill failed (10% weight loss). Other anodes performing satisfactorily with little attack on HSI and only slight surface softening on GR with backfill.
	1-HSI	2″x 60″	Hori.	Coke Breeze	0.43	2200	0.7	0.3	25,000	
	1-GR	3″x 60″	Hori.	None			0	0	21,000	
	6-GR	3″x 60″	Hori.	Coke Breeze			4.2	0.2	25,000	

HSI-High silicon iron.
HSI-Mo—High silicon iron containing molybdenum.
GR-Graphite.

change in the dimension of the anode. This installation was made along a lake approximately four years ago with the expectation that considerable moisture would be maintained in the bed. When the anode was partially uncovered, however, the soil was found to be compact and the bed exceedingly dry. These conditions actually have had very little effect on the performance of the anodes as very satisfactory service is being obtained. This application indicates the service life of high silicon iron anodes will far exceed the five year service life previously obtained from anodes of other material.

Ground bed installations using special backfill such as wood fiber plaster with and without a soil mixture continue to operate satisfactorily although the bed output is inconsistent as compared to similar installations where coke breeze backfill is used. An anode removed from a bed where a wood fiber plaster-soil backfill was used had a consumption rate of slightly less than 1 pound/amp yr after being in service approximately 3½ years. Though the cited consumption rate would provide a relatively long service life much lower rates have been obtained in a similar installation using coke breeze backfill.

High silicon iron anodes continue to function satisfactorily in an acid environment. This is outlined in Test No. 4, where the soil has a pH of approximately 3.2. These acidic conditions apparently have no deleterious effect on the high silicon iron because the output has remained constant for four years. Another material in a lesser period of time became ineffective and its output decreased over 90 percent.

Deep well installations (Test No. 26) are effectively utilizing high silicon iron anodes. Though installations of this type have not been in service as long as ordinary ground beds, reported data after two years of service show little or no change in operating characteristics and the beds are performing as designed. At this time it appears that the only limitation on this type of installation is the depth to which a rig is capable of drilling. The early deep well installations by one company were made with coke breeze backfill but it has since been learned that natural flake graphite is more desirable for the backfill because it does not have the tendency to bridge in the feed pipe as does the coke breeze. Reported advantages of such installations are: (1) Almost complete elimination of interference from other structures; (2) No engineering time required to locate proper soil; (3) Improved current distribution; (4) Ground beds can be located where conditions dictate; and (5) Very little if any expense is necessary to obtain a right-of-way. This method of protecting structures where applicable is being given considerable attention by corrosion engineers.

From the replies received in this year's survey it appears that the only limitation of high silicon iron anodes for ground bed installation is in chloride environments. Chlorides are often damaging and can cause selective attack and resultant failure especially if the chlorides become "trapped" at the anode and cannot escape. For such environments high silicon iron containing molybdenum should be used because of its superior resistance to chloride attack. However, the degree of superiority has not yet been established due to the limited numbers of installations that have had both alloys in service in the same environment. Eventually, Test No. 25 should provide a good comparison between the two materials under like conditions.

Fresh Water

Very little can be added to what has already been reported in past surveys pertaining to high silicon iron anodes in fresh water installations. Any detailed comments would be merely a duplication of previous information.

The general trend continues to show that standard high silicon iron performs very satisfactorily in fresh water applications (Table 2) at ambient temperatures with approximately 125 F being the upper temperature limit. For temperatures in excess of 125 F the molybdenum-containing alloy is recommended for an increased service life. Though a definite temperature limit has not been established for this alloy it appears that 160 F could likely be the upper limit. Of course, the composition of the water as well as the operating conditions are very important factors in establishing a maximum temperature limitation.

With the earliest high silicon iron fresh water installations now in service approximately five years and giving very satisfactory service it is apparent that this material has established itself as a very suitable material of construction for impressed current cathodic protection systems involving fresh water. Test No. 7 is a good example of an installation that has been in service for five years. Inspection of this anode showed only very minute pits at some areas. In fact they were so small that the weight loss of the anode was reported as zero. The grinding marks at the parting line of the anode were still bright and the manufacturer's trade mark was visible, all of which indicates many more years of service will be obtained.

Though 125 F is the recommended upper temperature limit for standard high silicon iron there are installations that exceed this temperature and still obtain satisfactory service. Test No. 12 illustrates this point. However, this is definitely considered an exception rather than the rule. Another exception but at the opposite extreme is Test No. 4. The temperature in this case is normal, but both the molybdenum-bearing and standard high

TABLE 2—Fresh Water Test Installations

Test No.	Anode Material	Anode Size	Water Conditions: Temp., Degrees F	Water Conditions: Resistivity	Total Current, Amperes	Average Current Density, Amp/Ft²	Approx. Amp. Hrs. Operation	Remarks
1	20-HSI	1″x120″	Normal	50,000	28–60	0.85	1,250,000	Ten HSI anodes in operation for 4 yrs. with very little weight loss. Number of anodes increased to combat selective corrosion areas on cathode. Aluminum required annual replacement.
	10-HSI	1″x 60″						
2	7-HSI	1″x 96″	Normal		3.3	0.21	168,000	Slight amount of corrosion after 4 yrs. No change in output. Aluminum lasted 14–18 mos. Equal protection from both materials.
	2-HSI	1″x168″			1.6	0.21		
3	6-HSI	1″x120″	Normal		13.4	0.80	369,000	3½ yrs. with no noticeable attack.
	4-HSI	1½″x 12″			1.3	0.80		
4	1-HSI-Mo	2″x 60″	Normal	1560	0.7	0.27		After 2 yrs. service HSI showed pitted condition (chlorine present). HSI-Mo replaced HSI and showed selective attack after 2 yrs. GR showed uniform attack. HSI passed considerably more current per anode. Treated water suspected of causing premature attack.
	26-GR	3″x 60″			12.5	0.12		
5	2-HSI	1″x120″	Normal	4830	1.1	0.20	34,800	No change in output or appearance in 46 mos.
6	1-HSI	1″x 60″	Normal		0.2	0.15	4,300	Test installation in service for over 3 yrs. with negligible corrosion. System now consists of 35, 1½″x12″ HSI anodes protecting water tanks.
7	1-HSI	2″x 60″	Normal	10,500–14,000	0.50–0.75	0.19–0.29	22,000	Five yrs. service with negligible corrosion.
8	4-HSI	1″x 90″	Normal	10,500–14,000	1.2–3.0	0.14–0.36	55,000	Over 4 yrs. service with consumption rates between 0.171–0.239 lbs./amp. yr./anode.
9	10-HSI	1″x180″	Normal		11.5	.28	260,000	Some anodes selectively attacked at gate area of casting. Fifty to sixty percent of anodes show no localized or general attack.
10	4-HSI	2″x 60″	70–168	3000–9000	7.8	0.75	200,000	Very slight weight loss on HSI after 39 mos., but two of the anodes have failed in the lead wire. Aluminum and impregnated graphite found unsatisfactory.
11	6-HSI	1″x 60″	Normal		1.6	0.19	42,500	No change in appearance or output after 38 mos.
12	6-HSI	1″x144″	150–200		5–8	.30–.48	155,000	Horizontally positioned anodes showed slight uniform corrosion after 34 mos.
13	1-HSI	1″x120″	Normal	5,000	1.0–2.4	0.72	125,000	Slight decrease in output after 4 yrs. protecting 30,000 gal. water tank. Very little corrosion indicates long service life.
14	3-HSI	2″x 60″	Normal	885	12.5	1.6	374,000	Consumption rate less than 0.055 lbs./amp. yr. after 41 mos. operation.
15	8-HSI	1″x360″	Normal	1500–4000	8.7–14.5	0.12–0.20	350,000	Three identical tanks having 12 anodes each show virtually no corrosion after 42 mos. almost 1,050,000 ampere hours total.
	4-HSI	1″x 60″						
16	48-HSI	1″x 60″	Normal		6.0	0.09	118,000	No change on 2 installations after 28 mos.
17	7-HSI	1½″x 12″	Normal		1.8–2.3	.62–.82	39,500	No apparent change after 34 mos. service.
18	4-HSI	1″x 60″	Normal		1.0–1.2	0.18	22,120	No change in appearance and output after 34 mos. on 50,000 gal tank.
19	54-HSI	1½″x 60″	Normal		85–135	0.80–1.26	2,695,770	Anodes showing slight uniform corrosion after 39 mos. in water tank. Output varies due to changes in water level.
20	27-HSI	1″x120″	115–140					HSI anodes at 140 F showed selective attack with service life of 18–36 mos. and all replaced with HSI-Mo. At 115 F and 130 F attack on HSI is slight and indicates much longer life. HSI with molybdenum being tested at 140 F and has shown no attack after 12 mos. No report in past year.
	9-HSI-Mo	1½″x120″	140					
21	3-HSI	1″x120″	170		4.2	0.50	100,000	Original HSI selectively attacked by this service after 1 yr. Replacement anodes made in HSI and HSI-Mo and after 1 additional yr. show a vast superiority for HSI-Mo. No change during 2nd year on HSI-Mo.
	3-HSI-Mo	1″x120″	170		4.2	0.50		
22	No additional information received during past year.							
23	1-HSI	1″x120″	Normal		1.2	0.43	30,000	No deterioration after 3 yrs. protecting 50,000 gal. water storage tank. Current output intentionally decreased.
24	14-HSI	1″x 60″	150		3–6	0.15–0.30	79,000	No noticeable attack after 26 mos. Number of anodes increased to 14 because 6 did not provide complete coverage.

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HSI—Mo—High silicon iron containing molybdenum.
GR—Graphite.

silicon irons showed selective attack. However, the presence of chlorine and other constituents in the treated water could be causing the selective attack and in the true sense of the word this installation probably should not be considered a fresh water installation. It is the only instance where high silicon iron anodes have not followed a definite pattern.

Connection failures have occurred on one of the early high silicon iron anode designs. This particular anode design is no longer in production, having been superseded by other designs with increased metal thickness at the connection area. This modification eliminated the problem.

Salt Water

The complete predictability of standard high silicon iron and the molybdenum containing alloy is still not achieved in all salt water (sea water and brackish) applications to the same extent as in ground bed and fresh water service. At this time it appears that 100 F should be the maximum temperature limit for the molybdenum-free alloy in salt water. Although this alloy has been used at temperatures in excess of 100 F, there are indications that between 100-125 F selective attack will sometimes occur. Above 125 F the standard high silicon iron is not recommended under any conditions because the rate of attack is highly unpredictable and generally excessive.

As previously reported the normal high silicon iron shows no superiority over other common anode materials in 100 percent sea water. However, as the resistivity increases when the sea water is diluted, high silicon iron shows a definite superiority. A paper presented by Tudor et al[1] points out the superiority of high silicon iron over graphite as resistivity increases. Tests No. 1 and 2 Table 3 are good examples of how high silicon iron

TABLE 3—Typical Salt Water Installations

Test No.	Anode Material	Anode Size	Environment Conditions: Temp., Degrees F	Environment Conditions: Resistivity	Total Current, Amperes	Average Current Density Amp/Ft²	Approx. Amp. Hrs. Operation	Remarks
1	40-HSI	3″x 60″	Normal	20–55	175	1.1	5,260,000	Forty-one mos. successful service protecting steel piling with no change in output. Installation has never been inspected. Horizontally installed both on hard bottom and suspended.
2	36-HSI	3″x 60″	Normal	55–250	265	1.84	8,900,000	Forty-six mos. protecting traveling screen on refinery raw feed water intake. Horizontally installed at various depths. Top anodes show uniform corrosion. Other anodes not inspected.
3	13-HSI	2″x 60″	Normal	50	90	2.66	2,000,000	Forty-three mos. protecting bulkhead. In horizontal position on stone fill (rip-rap). No inspection made to date. Current output more definitely established since original reports. Graphite anodes previously used. No report during past year.
4	14-HSI-Mo	12″ dia. hemi-sphere-oil	55–115	46	68	4.5	490,000	After two years service (over 1,500,000 amp. hrs.) HSI replaced with HSI-Mo because of heavy corrosion and inadequate protection. HSI-Mo performing satisfactorily after 10 mos. Anode redesigned to eliminate defects and increase service life.
5	18-HSI-Mo	1½″x 60″	170	Brackish	18	0.50	250,000	Some slight selective attack on HSI-Mo. Regular HSI failed in 2 mos. while HSI-Mo still suitable after 19 mos. No report in last 12 months.
6	12-HSI-Mo	3″x 60″	55–120	23	60	1.04	1,130,000	Three inch dia. HSI anodes originally used and provided a service life of 20-32 mos. Two inch dia. HSI corroding uniformly. Three inch dia. HSI-Mo substituted for HSI and are providing improved service after 12 mos.
	8-HSI	2″x 60″						
7	1-HSI	3″x 60″	Normal	Sea Water	15	3.8	141,800	HSI anodes at lower current density showed consumption rate of 0.64 lbs./amp. yr. while higher current density showed 1.85 lbs./amp. yr. after final 8-9 mos. test.
	1-HSI	3″x 60″	Normal	Sea Water	28	7.0	260,000	
8	18-HSI	1″x 60″	212	120	25–50	1.0–2.0	192,000	HSI anodes failed in 7 mos. due to corrosion by high temperature chlorine. Replaced with HSI-Mo. No additional information received during past yr. Refer to previous report.
9	36-HSI	1″x 60″	110	120	50–100	1.0–2.0	1,350,000	Anodes virtually unattacked, after 2 yrs. No further report during past year.
10	4-HSI	3″x 30″	40–75	75–5000	9	1.1	168,000	Anodes replaced after 25 mos. service. Corrosion predominantly at high temperature end of heat exchanger. Average consumption rate 0.232 lbs./amp. yr./anode. Replacement will eventually be made in HSI-Mo.
11	6-HSI	2″x 60″	65–150	28–8500	10	0.65	160,000	Crude oil in wash tanks believed to act as solvent on sealing compound causing failure of connection after 22 mos. Pitting of anodes attributed to chlorine gas. Treated graphite failed after 3 mos. HSI replaced with HSI-Mo.
12	29-HSI	2″x 60″	52–97	300–3000	10–90	.13–1.2		Twelve anodes showed varying degrees of selective attack: lead wire broke on 1 anode; 16 anodes showed little or no attack after 27 mos. Conditions too variable to approximate amp. hrs. of operation. Previously used graphite failed completely in like period.
13	6-HSI	2″x 60″	70	200	25	1.6	766,500	All anodes carefully weighed. After 3½ years high silicon iron consumed at 1.7 lbs./amp. yr. which caused failure of 3 anodes. After 2 yrs. HSI-Mo at 0.3 lbs./amp. yr., and treated graphite 1.52 lbs./amp. yr. Graphite anodes average 40% consumed.
	2-HSI-Mo	2″x 60″			9	1.7	158,000	
	2-GR	3″x 60″			10	1.25	175,000	

HSI—High silicon iron.
HSI—Mo—High silicon iron containing Molybdenum.
GR—Graphite

performs in salt water applications where the resistivity is higher than that encountered in 100 percent sea water. Increased current requirements and high temperatures (above 125 F) make the molybdenum-bearing high silicon iron the preferred alloy because of its better resistance in severe applications.

High silicon iron containing molybdenum will extend the temperature limitations of the molybdenum-free variety, but the maximum temperature limitation is still unknown. However, it is suspected that eventually this limitation will be found to be around 150-160 F and could possibly be altered somewhat depending upon the resistivity of the salt water and the current density requirements of particular installations.

The molybdenum containing alloy has replaced the molybdenum-free alloy in several installations known to be difficult for the latter alloy. Although the molybdenum containing alloy shows a definite superiority, it is also susceptible to attack though to a much lesser degree. This is illustrated by Test No. 13.

Throughout the investigational work on high silicon iron anodes no consideration was given to the type of covering being used on the anode leads. This fact should perhaps be considered in detail since there have been failures that have been attributed solely to this covering. In some of these instances replacement anodes were required because of the lead failure while the anodes were relatively free from attack.

Initially, high silicon iron anodes were constructed with the lead wire having high density polyethylene insulation and a polyvinyl chloride (PVC) outer jacket. In salt water applications the PVC jacket was attacked allowing the salt water to get between the outer jacket and the insulation. This resulted in severe blistering of the PVC and also allowed salt water to find its way into the connection resulting in failure. The high density polyethylene was not attacked by the salt water and this led to a covering that consisted entirely of high density polyethylene. This type of anode lead has the insulation applied in a single extrusion and the outside diameter is the same as the formerly used polyethylene insulation and PVC jacket. This type of cable is still being used today and there have been no failures reported.

Reference

1. S. Tudor, W. L. Miller, A. Ticker, and H. S. Preiser. Electro Chemical Deterioration of Graphite and High Silicon Iron Anodes in Sodium Chloride Electrolytes, Corrosion, 14, No. 2, 93t-99t (1958) February.

Corrosion Behavior of High Purity Vanadium*

By DAVID SCHLAIN, CHARLES B. KENAHAN and WALTER L. ACHERMAN

Abstract

Chemical and galvanic corrosion experiments at 35 C show that ductile vanadium is resistant to corrosion in substitute ocean water. It is also resistant in 60 percent sulfuric and 20 percent hydrochloric acids but corrodes rapidly in nitric acid solutions. Vanadium is less noble than stainless steel and copper and more noble than aluminum, magnesium and steel (SAE 4130) in substitute ocean water. 6.3.18

Introduction

VANADIUM PRODUCTION has been undertaken by several companies which are now making the metal on a limited scale in a form pure enough to be ductile. Vanadium has the physical and mechanical properties desirable in a structural metal, but this metal and alloys based on it have not been applied to industrial uses because of high production costs and a lack of information on their properties.

The Bureau of Mines is making an extensive study of vanadium and some of its alloys. A study of the corrosion properties of these metals is part of the program. This article gives the results of comparative studies of corrosion of ductile vanadium obtained from several sources.

Experimental Method

The studies included 6-, 15- or 30-day chemical and galvanic corrosion experiments at 35 C. Chemically pure grade chemicals, grade A helium (99.9+ percent pure), and standard commercial metals were used. The substitute ocean water used in this work was prepared according to a formula developed by the ASTM[1] with the following composition in gram per liter: NaCl, 24.53; $MgCl_2 \cdot 6H_2O$, 11.11; Na_2SO_4, 4.09; $CaCl_2$, 1.16; KCl, 0.695; $NaHCO_3$, 0.201; KBr, 0.101; H_3BO_3, 0.027; $SrCl_2 \cdot 6H_2O$, 0.42; NaF, 0.003.

Vanadium Specimens

Vanadium sheet used in these experiments was purchased from a commercial supplier or prepared in Bureau of Mines laboratories. Commercial vanadium was cold-rolled from 5/8-inch stock to finished sheet. Unannealed Bureau of Mines vanadium was 70-percent cold-rolled and had R_B hardnesses of 77 to 88; other strips were annealed 1 hour at 900 C in a vacuum of 5 x 10^{-5} mm Hg and had hardnesses of 62 to 70. Impurity contents of both types of vanadium are given in Table 1.

Vanadium specimens cut from commercial sheet for chemical corrosion experiments were 1 inch by 1 inch x 0.6 inch. Those cut from Bureau of Mines sheet were 2 inches by 0.5 inch by 0.06 inch. Galvanic couple specimens were 1/4-inch or 3/16-inch wide and 6 inches long. All specimens were cut and machined to 0.003 to 0.005 inch oversize and given a preliminary polish on a mechanical sander using 120-mesh abrasive cloth. Just before immersion, specimens were hand-polished with dry 3/0 emery cloth, rubbed with wet pumice, rinsed with water, dried, washed with carbon tetrachloride and weighed. At end of experiment, adherent corrosion products were removed by scraping the specimen with a firm rubber stopper. A chemical reagent was used when necessary.

Aeration

Some experiments were conducted with no forced flow of gas (natural aeration); others were conducted with a constant flow of helium or air through the corrodent. Before entering the corrosion apparatus, both gases passed through water to partially saturate them with water vapor. Air also was bubbled through 3 percent sodium hydroxide solution to remove carbon dioxide.

After this preliminary treatment, the helium or air was passed through the corrodent contained in one of the apparatus sets in Figures 1 and 2. The apparatus in Figures 1B and 2 consisted of a sealed bottle with a gas inlet and outlet and two water vessels. Incoming air or helium was passed through one water vessel to saturate the gas, with water vapor at 35 C, thus avoiding evaporation in the specimen vessel. Outgoing gas was passed through the other water vessel which served as a trap.

The gases were passed through the solution for about 1 hour before immersion of metal specimens and continuously throughout the experiment. Rates of flow, measured with rotameters, were approximately 250 ml per minute for both helium and air.

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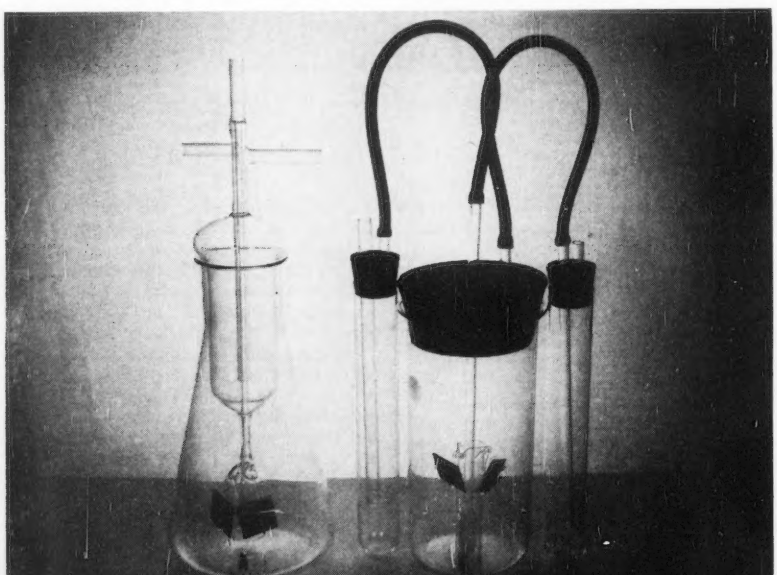

Figure 1—Apparatus used in chemical corrosion experiments. Flask A was used for ocean water test vessel and shows aerator-condenser assembly. Flask B was used for acid corrosion test vessel.

TABLE 1—Analysis of Vanadium, Percent

Source	C	O_2	H_2	N_2	Fe	Cr
Commercial Supplier...	0.035	0.06	0.0016	0.033	0.026	0.011
Bureau of Mines.....	....	0.062–0.107	....	0.004–0.03	0.21–0.48	0.02–0.06

TABLE 2—Vanadium in Substitute Ocean Water, 35 C, 30 Days[1]

Source of Vanadium	Corrosion Rate, MPY		
	Air	Natural Aeration	Helium
Commercial Supplier	0.2	0.4	0.0
Bureau of Mines[2]	0.2	...	0.0

[1] Number of replicates, 4.
[2] Annealed.

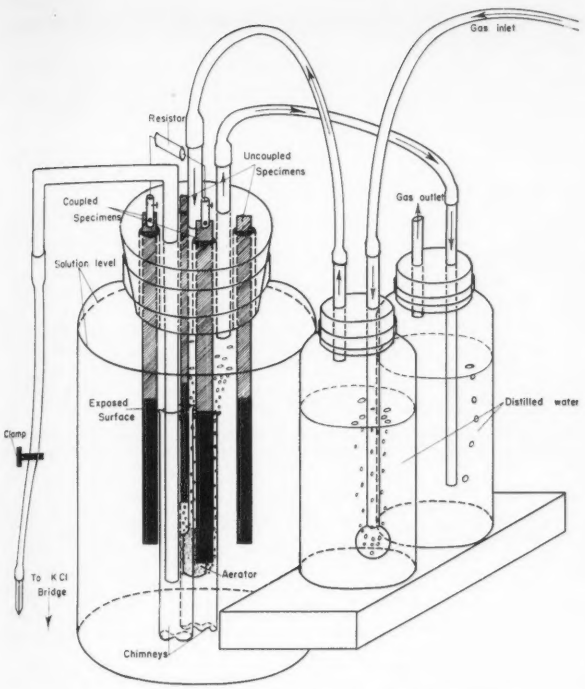

Figure 2—Apparatus used in galvanic couple corrosion experiments.

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TABLE 3—Vanadium[1] in Acid Solutions, 35 C, 6 Days

Source	Acid Concentration	Corrosion Rate, MPY	
		Air	He
Sulfuric Acid			
Commercial[2]	1.0N (4.8%)	0.6	0.6
Commercial[2]	3.1N (13.9%)	1.2	1.1
Commercial[2,3]	17.8N (58.8%)	3.9	3.9
Bureau of Mines, annealed	17.8N	3.0	3.2
Bureau of Mines, unannealed	17.8N	4.5	4.4
Hydrochloric Acid			
Commercial[2]	1.0N (3.6%)	0.6	0.5
Commercial[2]	2.0N (7.1%)	1.1	1.0
Commercial[2,3]	6.1N (20.2%)	5.5	5.5
Bureau of Mines, annealed	6.1N	5.1	4.3
Bureau of Mines, unannealed	6.1N	4.8	5.8
Nitric Acid			
Commercial[2]	.5N (3.1%)	1.0	0.8
Commercial[2]	2.0N (11.8%)	2.7	18, 6[4]
Commercial[2,3]	3.0N (17.2%)	98, 20[4]	19, 4470[4,5]
Bureau of Mines, annealed	3.0N	2270	2190[6]
Bureau of Mines, unannealed	3.0N	11, 40[4]	1350

[1] Number of replicates, 2, except as noted.
[2] These specimens (3 pairs) were previously used in substitute ocean water experiments. One pair of specimens was used for the three concentrations of each of the acids, starting with the least concentrated. The specimens were resurfaced before each experiment.
[3] Number of replicates, 4. Two specimens were used previously, as mentioned in note[2], and two specimens were fresh. No significant differences in the corrosion values for the 2 sets of specimens were noted in sulfuric and hydrochloric acids, and hence the 4 values were averaged in each instance. The values did vary in nitric acid.
[4] Corrosion rates for replicates do not agree and values are given separately.
[5] Experiment with fresh samples lasted 2 days.
[6] Experiment lasted 3.8 days.

Chemical Corrosion Method

Chemical corrosion experiments were conducted with the specimens completely immersed in 750 ml of corrodent. Two specimens were placed in each corrosion vessel. Apparatus used in substitute ocean water tests consisted of a 1-liter Erlenmeyer flask, a specimen support and an aerator-condenser assembly (Figure 1A.) Tests with acid solutions were conducted in a modified apparatus (Figure 1B). Loss in weight was used as a measure of corrosion.

Final acid concentrations were determined in experiments involving acid corrodents. Acid consumption was 5 percent or less in experiments with sulfuric and hydrochloric acids and with 0.5N nitric acid. For 2 and 3N nitric acid solutions, the acid consumption was sometimes as high as 35 percent.

Galvanic Corrosion Method

Galvanic couple experiments were conducted in the apparatus shown in Figure 2. The large bottle contained two specimens of each of the two metals involved in the experiment and 550 ml of solution. Specimens were inserted through slits in small rubber stoppers, which fitted tightly in holes in the large stopper. This formed gas-tight seals yet permitted external connections. Members of each couple were equal in exposed surface area. Upper portion of each specimen was insulated with rubber tape, leaving 3 inches of length exposed to the solution. Solution level in the specimen bottles was high enough to avoid solution-gas interface effects. Specimens were approximately 0.8 inch apart. One pair of dissimilar specimens was connected through a 1 ohm ($\pm$ 1 percent) resistor; the other two specimens were not coupled.

Galvanic currents flowing through the couples were determined by measuring voltage drops across the resistor and by applying Ohm's law. Voltage drops were measured with potentiometer and galvanometer. Galvanic corrosion rates were calculated from average galvanic currents by applying Faraday's law. Galvanic corrosion rates were averaged for the entire experiment. Weight losses of coupled and uncoupled metal specimens were determined and used in calculating total corrosion rates. Electrode potentials of each uncoupled specimen and of the couple were measured with a potentiometer, a saturated calomel electrode

TABLE 4—Corrosion of Vanadium (Commercial Supplier)—Metal Couples in Substitute Ocean Water, Flow of Air, 35 C, 15 Days

Second Metal	Anodic Metal	Galv. Corrosion, MPY (Calc. from Current)	Corrosion Rate, Vanadium, MPY (Based On Weight Loss)		Corrosion Rate, Second Metal MPY (Based On Weight Loss)	
			Coupled	Uncoupled	Coupled	Uncoupled
Stainless Steel, Type 316	.....	0.0	0.8	0.8	0.0	0.0
Stainless Steel[1], Type 316	.....	0.0	0.2	0.1	0.0	0.0
Copper	Vanadium	2.0	1.2	0.5	0.0	0.9
Copper[1]	Vanadium	2.0	0.7	0.2	0 0	0.5
Aluminum	Aluminum	0.3[2]	0.0	0.5	0.1	0.0
Steel SAE 4130	Steel SAE 4130	3.0	0.05	0.7	9.5	4.7
Magnesium	Magnesium	353	0.0	0.3	727	35.2
Magnesium[1,3]	Magnesium	378	0.0	0.9	920	41.5

Note: Steady-state electrode potential values for single metal specimens: magnesium, ca —1.4; aluminum, —0.59; steel SAE 4130, —0.45; vanadium, —0.13; stainless steel, —0.1 to +0.18; copper, 0.04.
[1] Experiments were done with unannealed Bureau of Mines vanadium. Other experiments were done with vanadium purchased from commercial supplier.
[2] Galvanic current during first 900 minutes was equivalent to average corrosion rate of 6.3 mpy; during remainder of experiment, galvanic current was negligible.
[3] Experiment lasted 6 days.

and a saturated potassium chloride bridge. The apparatus was arranged to measure the potential of the whole specimen rather than that for a particular point. Electrode potentials are reported on the standard hydrogen electrode scale.

Results of Experiments

Vanadium, according to results obtained from these experiments, is resistant to corrosion in substitute ocean water saturated with air and is completely resistant in the absence of air (Table 2). It is resistant to corrosion in sulfuric and hydrochloric acids, even in rather concentrated solutions (Table 3). In these environments, corrosion rates are not affected by absence of air. Vanadium resists corrosion in 0.5N nitric acid but corrodes in 3N acid (Table 3). Data for 3N nitric acid solutions are erratic.

When corrosion rates for replicates are in substantial disagreement, the values are given separately in the table. There are no significant differences between the corrosion rates for vanadium obtained from a commercial supplier and for annealed and unannealed vanadium prepared in Bureau of Mines laboratories. Corrosion rates published by Kinzel[2,3] pertain to different temperatures and acid concentrations but are consistent with data presented here.

Vanadium is more corrosion resistant than titanium in some sulfuric and hydrochloric acid solutions. For example, titanium corrodes at rates of 31, 341 and 43 mpy in 10, 40 and 60 percent air-saturated sulfuric acid solutions, respectively, and at 183 mpy in 20 percent air-saturated hydrochloric acid solution.[4] In the absence of air, titanium also corrodes in 5 percent sulfuric (18 mpy) and 4 percent hydrochloric (11 mpy) acid solutions.[5]

In galvanic corrosion experiments, electrode potentials of the single metal specimens and of the couple generally reached steady-state values soon after immersion. Steady-state electrode potential values for the single specimens are given in the note in Table 4. In substitute ocean water with a flow of air, the single electrode potential of vanadium was less noble than those of stainless steel or copper. When coupled with steel or copper, vanadium always was the anodic (corroding) member of the couple. However, the vanadium-stainless steel couples did not generate detectable galvanic currents, and corrosion rates of vanadium and stainless steel were not changed significantly as a result of contact with each other. Vanadium-copper couples generated small galvanic currents. The normally low corrosion rate of each metal was changed by the contact; vanadium corroded a little faster and the corrosion of copper was eliminated.

Vanadium was more noble in this environment than aluminum, magnesium or SAE 4130 steel. In the vanadium-aluminum couple, the initial galvanic current was large but dropped to zero during the first 15 hours. Corrosion of vanadium was eliminated as a result of contact with the aluminum; aluminum corroded very slightly because of the contact. The surface of the coupled aluminum specimen acquired numerous small pits. The vanadium-steel SAE 4130 couple generated a galvanic current equivalent to about 3 mpy. Corrosion of vanadium was decreased and corrosion of steel was doubled as a result of the coupling. Vanadium-magnesium couples generated very large currents, equal to corrosion rates of more than 350 mpy. Corrosion of vanadium was eliminated and the corrosion of magnesium was greatly increased as a result of contact.

Vanadium purchased from a commercial supplier and vanadium produced in Bureau of Mines laboratories behaved in a similar manner in galvanic couples. The behavior of vanadium and the other metals is consistent with their relative electrode potentials as given in the note in Table 4.

Conclusions

Vanadium is resistant to corrosion in substitute ocean water at 35 C. It is resistant to corrosion in 60 percent sulfuric acid and 20 percent hydrochloric acid at 35 C. Vanadium resists corrosion in 3 percent nitric acid solution, corrodes slowly in 12 percent acid and corrodes very rapidly in 17 percent acid. Absence of air has little or no effect on the corrosion of vanadium in these environments.

In substitute ocean water vanadium is less noble than stainless steel and copper and more noble than aluminum, magnesium or SAE 4130 steel. Coupled with these metals, vanadium behaves as a sacrificial metal for copper but is protected by aluminum, SAE 4130 steel and magnesium. Neither vanadium nor stainless steel is affected by contact with each other.

Vanadium from the two sources exhibited essentially the same corrosion properties although there was a considerable difference in iron content. Annealed and unannealed vanadium exhibited similar corrosion characteristics.

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3. William Rostoker. Metallurgy of Vanadium. John Wiley & Sons, Inc., 1958, p. 134.
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Any discussion of this article not published above will appear in the December, 1960 issue.

High-Temperature, High-Pressure Oxidation Of Alloys Caused by Carbon Dioxide*

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Abstract

Presents data on corrosion of heat resistant alloys by high pressure carbon dioxide. Describes oxidation behavior of 26 alloys in 1000 to 2000 psi carbon dioxide in temperature range of 1100 to 1740 F. Oxidation rates of heat resistant alloys in 2000-psi carbon dioxide at 1300 and 1500 F were very low. In all cases, scales were protective. Oxidation followed a parabolic or slower rate law. At 1740 F, most alloys tested exhibited excessive scaling in 1000-psi carbon dioxide. 3.4.6

Introduction

GAS-COOLED reactors are currently being designed with outlet temperatures in the range 1200 to 1500 F and maximum cladding temperatures on the fuel element approaching 1800 F. Many gases have been considered for use as coolants for this type of reactor, and each offers a unique set of materials problems peculiar to the chemical nature of the coolant gas, its temperature, and pressure. This article reports some of the results obtained during a coolant evaluation program conducted on the Maritime Gas-Cooled Reactor project, and describes the oxidation of reactor materials in high pressure, high temperature carbon dioxide.

Materials Selected for Corrosion Testing

MGCR materials for such applications as fuel and moderator cladding, piping, turbine blades, etc., show a broad range of corrosion resistances and high temperature strengths. They extend from the carbon-molybdenum steels of relatively low oxidation resistance through the chromium-molybdenum grades (in increments of chromium content), to the ferritic stainless steels. For the severest conditions, a number of austenitic steels and nickel base alloys were evaluated. In addition, two iron-chromium alloys containing aluminum and a cobalt base alloy were included. Table 1 presents compositions of the materials tested. Properties of many of these alloys, their fabricability and their response to irradiation have been summarized.[1] The high temperature strengths of some of these alloys are shown in Table 2.

These materials were tested in a static carbon dioxide system at a pressure of 2000 psi and temperatures of 1300 and 1500 F and at a pressure of 1000 psi and temperatures of 1100 and 1740 F.

Dependence of Corrosion on Time and Temperature

Time

A number of relationships between oxidation and time have been found empirically. Several rate equations are obeyed over different regions of oxide thickness at any given temperature. In general, logarithmic, parabolic and linear equations give the best analytical representation of oxidation data, although cubic rates are also encountered. These equations are

$$y = k_1 \ln (1 + t/t_o) \text{ (logarithmic)} \quad (1)$$

$$y^2 = k_2 t + s(T) \text{ (parabolic)} \quad (2)$$

$$y = k_3 t + b(T) \text{ (linear)} \quad (3)$$

$$y^3 = k_4 t + c(T) \text{ (cubic)} \quad (4)$$

where y is the metal oxidized, k is the rate constant, t is time, and t_o, s(T), b(T), and c(T) are constants dependent on temperature and oxygen pressure.

In some cases, the logarithmic rate is followed during the initial stages of oxidation to a film thickness of a few hundred angstroms; beyond this thickness the process is diffusion controlled and the parabolic rate holds. The linear rate operates when the film is porous or volatile.

When the rate of oxidation is greater than that required for the parabolic law to operate but less than that required for the linear law, there is probably a diffusion process involved, overlapped by a reaction due to pores forming in the film or to evaporation of the film.

Temperature

Temperature variation of the oxidation-rate constant is expressed by the Arrhenius relation $k = A \exp(-Q/RT)$, where A is the frequency factor which contains an entropy term and Q is the activation energy. Occasionally, a plot of log k versus the reciprocal of the absolute temperature gives two intersecting straight lines, indicating the two different mechanisms determine the rates at low and high temperatures, namely,

$$k = A' \exp(-Q'/RT) + A'' \exp(-Q''/RT). \quad (5)$$

Temperature also governs the extent to which the different rate laws are followed. Oxidation of some metals follows the logarithmic rate at low temperatures, the parabolic rate at intermediate temperature and the linear rate at high temperatures. These temperature ranges depend on the properties of the oxides formed. For example, if a more stable film is formed at higher temperatures, the rates may actually decrease as the temperature is increased.

Carbon Dioxide Corrosion Tests

Screening tests were conducted in 2000-psi static carbon dioxide at 1300 and 1500 F to rank the possible materials according to the following factors:

1. How rapidly they oxidized. This was accomplished by determining the time dependence of the weight gain and noting how effective the corrosion products were in protecting the materials from further oxidation, as was discussed previously.

2. Manner in which corrosion proceeded. Metallographic investigations determined whether the oxidation resulted in a uniform reduction in specimen thickness or was of localized intergranular or pitting type. The latter two deleterious types of atack are not revealed in weight-gain data.

3. Adherence of the scale. The scale was abraded with a hard steel stylus while being examined under a microscope. A scale was ranked "adherent" if it could not be scraped off.

4. Compositions of the scales. These were determined by x-ray diffraction measurements.

Most corrosion samples used were cut from annealed sheet stock 0.032 to 0.050 inch thick and had surface areas varying from 10 to 20 cm^2. Samples were prepared by shearing a specimen from annealed sheet, drilling two holes for support, degreasing, washing with distilled water and drying with acetone. Specimens were hung on a Type 310 stainless steel rack (single-point contact) and separated by small quartz spacers. Testing vessels were 5-inch ID autoclaves, 24 inches deep. Nichrome wound tubular heater elements were placed inside the vessel and insulated from the wall. Before use, vessels were outgassed at 1500 to 1600 F under vacuum.

After being outgassed, the vessels were cooled and the samples were inserted. The vessel was then closed and evacuated, filled with carbon dioxide and reevacuated at least three times. Finally, the vessel was charged with carbon dioxide at about 850 psi. Heating to the test temperature resulted in a pressure buildup to 2000 psi. Periodically the specimens were removed, weighed and replaced for further exposure. From four to eight specimens of each material were exposed in each test, permitting removal of a sample after various exposures for metallographic and x-ray diffraction studies. Specimens were plated with nickel before being mounted and polished to facilitate metallographic examination.

Grade-B, medical-grade carbon dioxide was used in initial tests and welding grade in later tests. These grades are 99.5 and 99.9 percent pure, respectively. No differences in corrosion rates were noted between the two carbon dioxide grades.

Because the system is static, carbon

★ Submitted for publication April 16, 1959. A paper titled "Oxidation of Metals by Carbon Dioxide at Temperatures of 1100 F to 1740 F and Pressures of 1000 to 2000 psi."

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TABLE 1—Structural and Cladding Materials Selected for Evaluation

Alloy Type	Designation	Composition (%)									
		C	Mn	P	S	Si	Ni	Cr	Mo	Cb	Other
Austenitic Stainless Steels	330	0.08	2.12	0.012	0.006	0.69	14.34	34.69	....	....	
	310	0.07	1.83	0.02	0.019	0.65	19.62	24.25	....	....	
	309	0.08	1.61	0.018	0.015	0.53	13.96	22.70	....	....	
	316	0.08	1.51	0.02	0.02	0.51	10.98	17.43	2.43	....	
	347	0.05	1.43	0.024	0.009	0.63	9.42	17.99	....	0.81	
	304	0.08	1.47	0.023	0.018	0.57	10.11	18.83	....	....	
Ferritic Alloys	446	0.07	0.62	0.027	0.024	0.40	0.29	24.87	....	....	
	430	0.05	0.46	0.014	0.010	0.33	....	15.56	....	....	
	405	0.05	0.40	0.025	0.019	0.56	....	12.64	....	....	Al, 0.26
	DB-2	....	....	....	....	....	....	5.0	....	1.0	Ti, 0.5; Al, 7.0 (nominal)
	FeCrAl	....	....	....	....	....	....	25.0	....	....	Al, 5.0 (nominal)
	9 Cr, 1 Mo	0.12	0.37	0.010	0.009	0.30	....	8.60	1.07	....	
	5 Cr, ½ Mo	0.11	0.37	0.012	0.011	0.41	....	4.60	0.53	....	
	2¼ Cr, 1 Mo	0.10	0.44	0.019	0.012	0.26	....	2.26	0.98	....	
	1¼ Cr, ½ Mo	0.12	0.45	0.012	0.023	0.65	....	1.20	0.52	....	
	C, Mo	0.12	0.42	0.014	0.016	0.21	....	....	0.54	....	
Nickel-base Alloys	"A" Nickel	0.10	....	....	....	....	99.4	....	....	....	Cu, 0.10; Fe, 0.15 (nominal)
	Inconel	0.04	0.21	....	0.007	0.19	76.80	15.46	....	....	Cu, 0.06; Fe, 6.76
	Inconel X	0.05	0.56	....	0.007	0.31	72.77	15.32	....	1.01	Cu, 0.08; Al, 0.71; Ti, 2.47; Fe, 6.69
	Nichrome V	0.05	0.15	....	....	0.39	Bal.	19.56	....	....	Fe, 0.14; Al, 0.01
	Hastelloy B	0.02	0.76	0.008	0.017	0.64	Bal.	0.42	27.17	....	Fe, 5.01; V, 0.37
	Nimonic 80	0.05	....	....	....	....	76	20	....	....	Ti, 2.3; Al, 1.0; Fe, 0.5 (nominal)
Miscellaneous Alloys	Haynes 25	0.10	1.48	0.009	0.009	0.86	10.09	19.91	....	....	W, 15.31; Fe, 2.24; Co, Bal.
	Niobium	....	....	....	....	....	....	....	....	100	Nominal
	Nb, 1 Zr	....	....	....	....	....	....	....	....	99	Zr, 1.0 (nominal)
	Nb, 10 Mo, 10 Ti	....	....	....	....	....	....	....	10	80	Ti, 10 (nominal)

TABLE 2—High Temperature Mechanical Properties of Some Alloys Tested

Alloy Type	Temp. (F)	Short-Time Tensile Strength (psi)	Rupture Strength (psi)		Creep Strength (psi)	
			10^3 hr.	10^4 hr.	1% in 10^4 hr.	1% in 10^5 hr.
316	1,300	46,000	17,500	12,000	7,900	3,800
	1,500	27,500	6,600	3,400	2,800	1,300
Inconel	1,300	.....	.....	.....	6,200	4,000
	1,500	.....	3,700	.....	1,500	700
309	1,300	44,000	12,700	7,800	4,800	2,100
	1,500	27,000	4,500	2,700	1,000	400
446	1,300	17,000	2,700	1,600	700	300
	1,500	8,000	1,200	800	....	....
430	1,300	15,500	2,800	2,200	1,400	900
	1,500	7,000	900	500	....	....

TABLE 3—Weight Change of Chromium Steels in 1000-psi, 1100 F Carbon Dioxide

Material	Weight Change (mg/cm²)*	
	136 hr.	591 hr.
C, ½ Mo	43.6	...
1¼ Cr, ½ Mo	25.4	...
2¼ Cr, 1 Mo	8.12	...
5 Cr, ½ Mo	5.72	25.2
9 Cr, 1 Mo	5.27	26.9

* Average of four samples.

monoxide builds up as a result of the following reactions:

$$2\,CO_2 \rightarrow 2\,CO + O_2 \qquad (6)$$

$$xO_2 + yM \rightarrow MyO_2x \qquad (7)$$

Figure 1 shows the temperature and pressure dependence of the first equilibrium. A gas sample was analyzed at the end of each run. During the first 100 hours of oxidation, carbon monoxide increased from 0 to between 0.5 and 0.6 vol-percent. In later runs, after the initial oxide films were formed, carbon monoxide build-up was not so large, ranging from 0.2 to 0.3 vol-percent.

Carbon Dioxide Corrosion Test Results

From an engineering standpoint, oxidation rates of the alloys tested in 2000-psi carbon dioxide at 1300 and 1500 F are encouraging. In most cases, the scales formed are protective (to > 1600 hr), and scaling in only one case was observed. Data for representative alloys from each group (Figures 2 through 8) indicate very low oxidation rates which decrease with time. (Two different abscissas were used in the figures: 0.1 to 10 and 0.01 to 1.0 mg/cm².) Reference slopes are shown on the curves in the lower right-hand corner for comparison. Slopes for all the alloys tested are indicative of parabolic (diffusion-controlled) or smaller oxidation rates. Figures 9 through 14 are graphs of typical specimens of each alloy family drawn on the same scale for comparison. The right-hand scale on Figures 9 through 14 is for penetrations, as calculated for the case of uniform oxidation.

Among the various alloys tested, there was considerable overlap in corrosion resistance (Figures 9 through 14). For example, many austenitic and ferritic stainless steels and nickel-base alloys fell together. Slopes for the austenitic steels consistently were smaller than those for the ferritic materials and the nickel base alloys. However, the most oxidation-resistant materials (those that showed the smallest weight gain) were ferritic or nickel base. In any event, corrosion rates observed in the alloys tested at 1300 and 1500 F were small, many being less than 0.001 ipy.

Niobium and the two niobium base alloys disintegrated in less than 100 hours in 2000-psi, 1300 F carbon dioxide.

The temperature dependence of the heat-resistant alloys studied is given in Figures 15, 16 and 17. Only a limited number of data were obtained at 1100 and 1740 F, and they indicated that many materials which showed good performance at the lower temperature no longer resisted scaling in 1000-psi, 1740 F carbon dioxide. Types 430, 316, 309 and 310 stainless steel and Inconel X showed weight losses after a 500-hour exposure. At 1100 F the oxidation resistance of the nickel base alloys and the stainless steels was excellent, but the chromium-molybdenum piping materials oxidized rapidly (Table 3). At this temperature the piping will necessarily be stainless steel, probably Type 316.

In any corrosion study, the nature of the surface being corroded should be specified. In these screening tests, all materials were tested in the as-received condition. The only surface preparation of the specimens was cleaning the surfaces by removing any foreign compounds which might have been picked up before testing.

The importance of surface conditions is reflected in Figure 4, which shows the oxidation behavior of Type 430 stainless steel with two types of surfaces. The specimen group represented by the upper 1500 F band was pickled before testing; the lower group was degreased only and had mirror-like surfaces. These data indicate the sensitivity to surface condition and point out the importance of using specimens whose surface conditions are realistically similar to those encountered on the materials used in reactor components.

Attack during oxidation can occur in several ways. The most desirable is one in which a uniform, thin, adherent surface oxide is formed which grows slowly

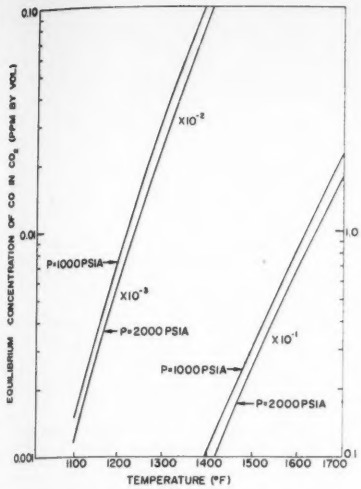

Figure 1—Decomposition of carbon dioxide ($CO_2 = CO + \frac{1}{2} O_2$)

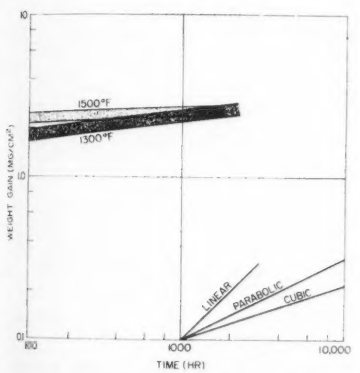

Figure 2—Weight change with time of Type 304 stainless steel exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

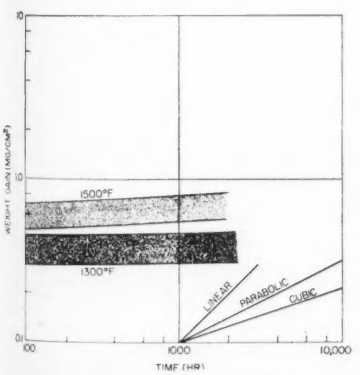

Figure 3—Weight change with time of Type 310 stainless steel exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

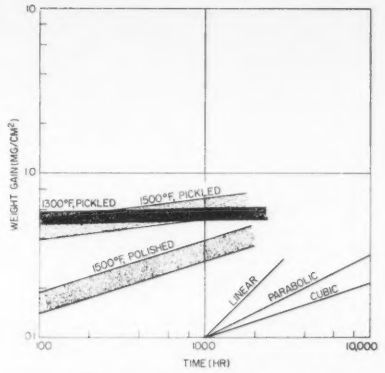

Figure 4—Weight change with time of Type 430 stainless steel exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

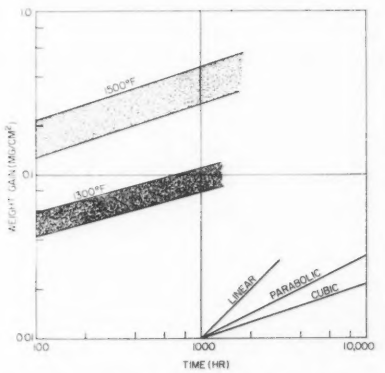

Figure 5—Weight change with time of Type 446 stainless steel exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

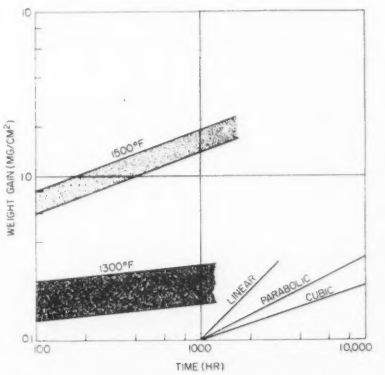

Figure 6—Weight change with time of Inconel X exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

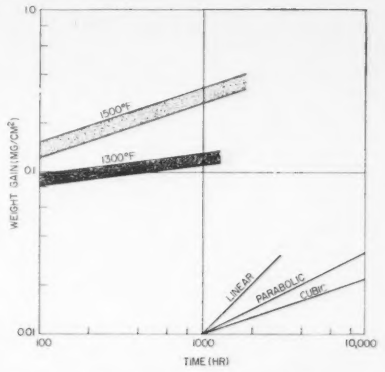

Figure 7—Weight change with time of Nichrome V exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

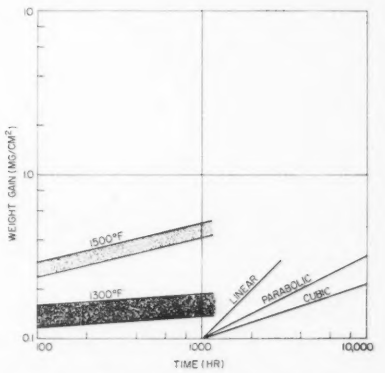

Figure 8—Weight change with time of Haynes 25 alloy exposed to 2000-psi carbon dioxide at 1300 and 1500 F.

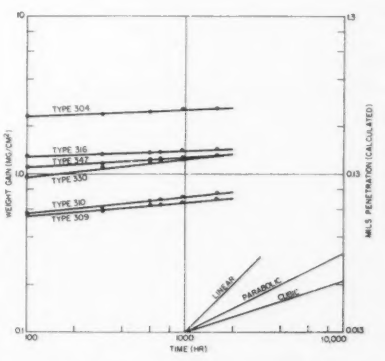

Figure 9—Comparison of weight changes with time of austenitic stainless steels exposed to 2000-psi, 1500 F carbon dioxide.

in thickness. In this case, the weight-gain data can be converted to penetration rates if the composition of the scale is known. Unfortunately, other types of attack also are possible. Localized attack can occur which results in severe pitting or preferential attack at grain boundaries. These two latter attacks can result in rapid corrosion failure, even when overall oxidation rates, as measured by weight gain, are low. It is essential, therefore, that corrosion specimens be sectioned and examined metallographically to determine the nature of the attack.

Results of metallographic examination of samples exposed at 1500 F are summarized in Table 4. The penetration listed is the maximum penetration depth observed on two cross sections of a single corrosion sample. The type of attack is noted in the last column. Figure 18 shows typical photomicrographs of the types of oxidation discussed above. Note that some of the materials that show low weight-gain rates and have very protective scales show relatively high penetrations, because of either intergranular oxidation or pitting-type attack. Localized attack seems most likely to occur in the austenitic stainless steels and in the nickel base alloys.

In addition to corrosion attack, metallographic examination revealed the grain growth characteristics of the alloys. All grain growth noted occurred in the ferritic steels. Resistance to grain growth is important because it helps determine an

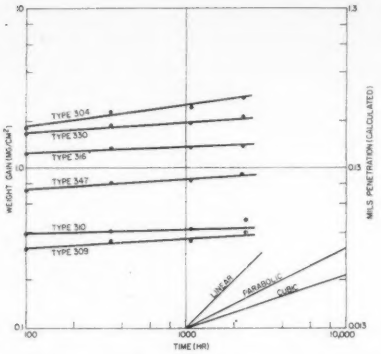

Figure 10—Comparison of weight changes with time of austenitic stainless steels exposed to 2000-psi, 1300 F carbon dioxide.

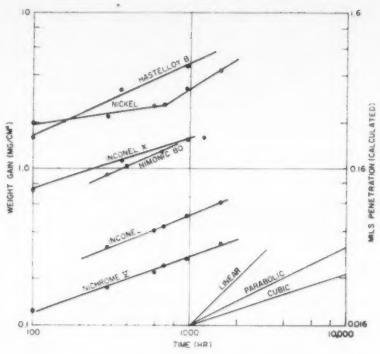

Figure 13—Comparison of weight changes with time of nickel base alloys exposed to 2000-psi, 1500 F carbon dioxide.

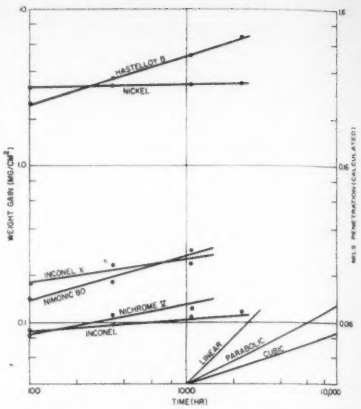

Figure 14—Comparison of weight changes with time of nickel base alloys exposed to 2000-psi, 1300 F carbon dioxide.

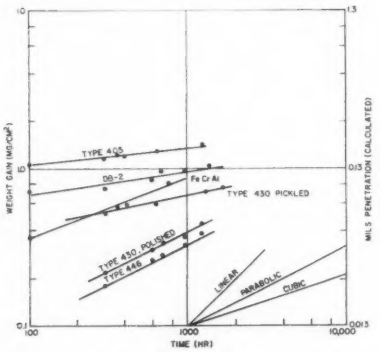

Figure 11—Comparison of weight changes with time of ferritic alloys exposed to 2000-psi, 1500 F carbon dioxide.

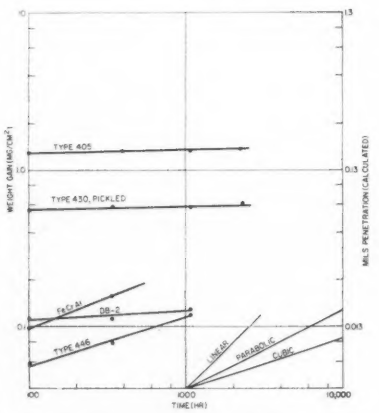

Figure 12—Comparison of weight changes with time of ferritic alloys exposed to 2000-psi, 1300 F carbon dioxide.

TABLE 4—Metallographic Results of Alloys Exposed to 1500 F, 2000-psi Carbon Dioxide

Material	Time (hr)	Maximum Penetration (Mils)	Grain Growth	(%) Second Phase	Remarks
430	1565	0.15	8X	5	Uniform oxide
446	1565	0.15	8X	15	Massive second phase
405	1265	0.8	2X	10	Oxide "fingers"
FeCrAl	1265	0.5	2X	5	Uniform oxide
DB-2	1565	0.2	...	7	Uniform oxide
304	1565	2.0	...	2	Pits
309	1565	0.4	...	22	20% sigma
310	1565	2.5	...	22	20% sigma
347	1565	0.75	...	3	
316	1565	0.6	...	10	8% sigma
330	1565	1.4	...	3	Slight pitting
Nickel	1565	7.0	...	0	Grain boundary oxide
Hastelloy B	970	1.0	...	10	.....
Nimonic 80	970	1.4	...	10	.....
Inconel	1565	1.3	...	1	Intergranular oxide
Inconel X	1265	1.3	...	1	Intergranular oxide
Nichrome V	1565	2.5	...	5	Pits
Haynes 25	970	0.2	...	..	Uniform oxide

TABLE 5—Composition of Scales Formed on Materials in 2000-psi, 1500 F Carbon Dioxide

Material	Scale Components	Remarks*
330	(Spinel)s	A
310	(Spinel)s; ([Cr, Fe]$_2O_3$)w	A
309	(Spinel)s; ([Cr, Fe]$_2O_3$)w	A
316	(Spinel)s	A
347	(Spinel)s	A
304	(Spinel)s	A
19-9DL	(Spinel)s; ([Cr, Fr]$_2O_3$)w	A
446	(Spinel)s; ([Cr, Fe]$_2O_3$)w	B
430	(Spinel)m; (Cr_2O_3)w	B
405	(Spinel)s	A
DB-2	(Spinel)s	A
FeCrAl	(Spinel)w	A
Nickel	(NiO)s	A
Inconel	(NiO)s; (Spinel)m ([Cr, Fe]$_2O_3$)w	B
Nichrome	(NiO)m; (Spinel)w; (Cr_2O_3)w	A

* A = Very adherent
B = Moderately adherent

alloy's ability to perform well mechanically at high temperature.

This is an important point because ferritic materials are the only alloys which can operate unprotected in contact with graphite at 1500 F without carburizing.[7] However, they cannot support any significant load in the high temperature regions of the reactor core.

X-ray diffraction patterns taken from the surface of corrosion specimens exposed at 1500 F gave a qualitative analysis of the compounds in the scales formed in carbon dioxide. The compounds formed on the iron and nickel base alloys were oxides of chromium, iron and nickel and the spinels. The spinels and Fe_3O_4 have the same structure and are mutually soluble, as are Cr_2O_3 and Fe_2O_3. This makes definite identification by x-ray diffraction very difficult.

Table 5 lists the compounds present in the scales for each material. Some patterns obtained were characteristic of more than one spinel and can be interpreted as coming from specific spinels or from solid solutions. The small letters s, m, or w indicate the relative intensity of the pattern—strong, medium, or weak. The scales formed are in many cases similar to those formed in air or oxygen.[2]

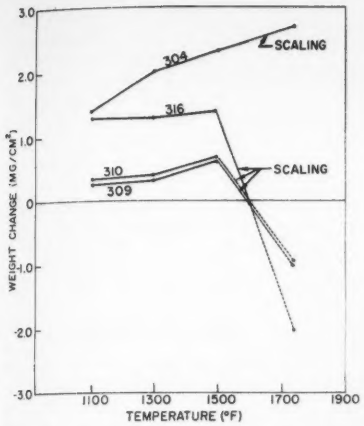

Figure 15—Weight change in 500 hr versus temperature for austenitic stainless steels exposed to carbon dioxide (1100 and 1740 F data are at 1000 psi; 1300 and 1500 F data are at 2000 psi).

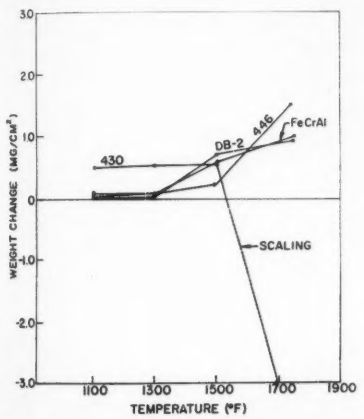

Figure 16—Weight change in 500 hr versus temperature for ferritic alloys exposed to carbon dioxide (1100 and 1740 F data are at 1000 psi; 1300 and 1500 F data are at 2000 psi).

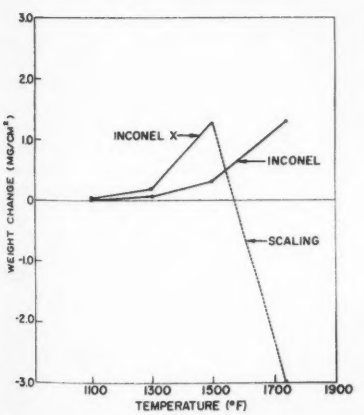

Figure 17—Weight change in 500 hr versus temperature for nickel base alloys exposed to carbon dioxide (1100 and 1740 F data are at 1000 psi; 1300 and 1500 F data are at 2000 psi).

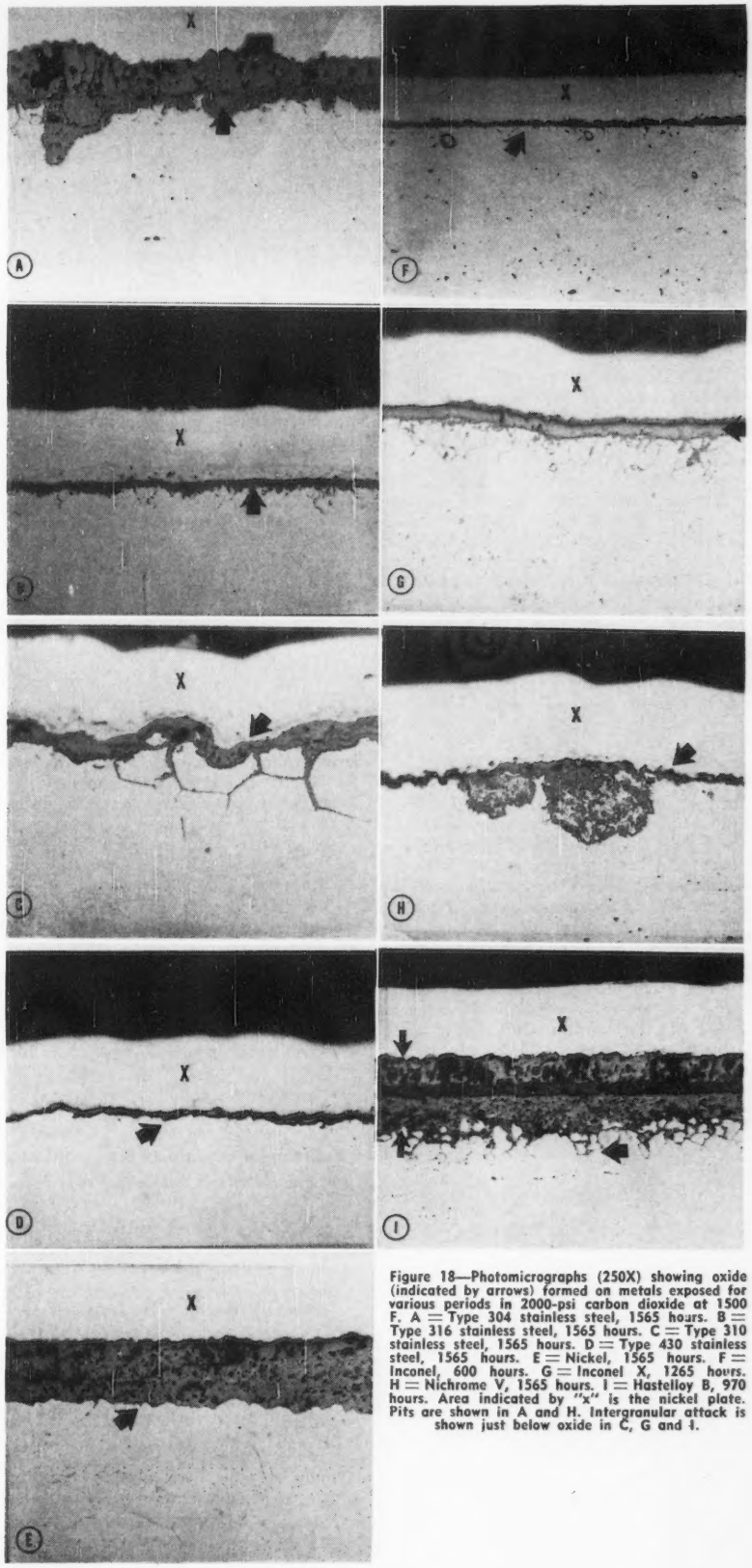

Figure 18—Photomicrographs (250X) showing oxide (indicated by arrows) formed on metals exposed for various periods in 2000-psi carbon dioxide at 1500 F. A = Type 304 stainless steel, 1565 hours. B = Type 316 stainless steel, 1565 hours. C = Type 310 stainless steel, 1565 hours. D = Type 430 stainless steel, 1565 hours. E = Nickel, 1565 hours. F = Inconel, 600 hours. G = Inconel X, 1265 hours. H = Nichrome V, 1565 hours. I = Hastelloy B, 970 hours. Area indicated by "x" is the nickel plate. Pits are shown in A and H. Intergranular attack is shown just below oxide in C, G and I.

TABLE 6—Comparison of Oxidation of Various Stainless Steels in Air and Carbon Dioxide

Type	168 hr.		504 hr.		816 hr.		1008 hr.	
	Air	CO_2	Air	CO_2	Air	CO_2	Air	CO_2
Weight Gain at 1500 F (mg/cm²)								
17 Cr	0.48	0.50	0.75	0.58	0.85	0.62	0.99	0.65
27 Cr	0.4	0.24	...	0.35	...	0.43	1.0	0.45
18-8	0.35	2.2	0.62	2.5	0.76	2.5	0.85	2.5
18-8 Cb	0.32	1.2	0.64	1.2	0.82	1.25	1.1	1.3
18-8 Mo	0.57	1.3	0.84	1.4	5.0	1.4	5.0	1.45
Weight Gain at 1740 F (mg/cm²)								
27 Cr	1.9	0.85	...	1.5	...	...	...	...
18-8	24	1.8	25	2.5	...	...	...	...
18-8 Mo	7.2	1.6	7.5	2.0	...	...	...	...

Scales designated as very adherent in Table 5 withstand severe abrading with a hardened steel stylus without flaking. Those designated as moderately adherent cannot be removed by vigorous rubbing but can be removed with the stylus. These tests were conducted on specimens exposed for 1565 hours at 1500 F. The scales on Inconel, Type 446 and Type 430 were very thin and ruptured under the stylus test. They are rated (possibly too conservatively) as only moderately adherent.

A comparison of alloy behavior in high pressure carbon dioxide with alloy behavior in high temperature atmospheric air[3] is given in Table 6 for 1500 and 1740 F. High pressure carbon dioxide is less corrosive than air at 1740 F and much the same as air at 1500 F.

Many of the stainless steels evaluated in these tests are likely to form the sigma phase in the temperature range involved. Sigma in the iron-chromium system is a hard and brittle compound of iron and chromium having a composition between about 43 and 50 atom-percent chromium. It is a low temperature crystalline form existing at temperatures below about 1500 F.

The exact composition and temperature ranges where sigma is found vary widely in the stainless steels. Sigma phase effects are not large at high temperatures but can lead to embrittlement at low temperatures. The percentage of second phase listed in Table 4 refers in most cases to sigma; however, some alloys contain carbides and other compounds which precipitate. These percentages are the result of visual estimation.

Conclusions

The results of the screening tests can be interpreted as follows:

1. Weight-gain data, a measure of gross oxidation rates, revealed that all heat resistant alloys tested at 1300 and 1500 F oxidized slowly enough to be considered for use in the MGCR; i.e., all of the scales are protective.

2. Metallographic examination, however, revealed that certain materials were subject to localized attack: namely, pitting and intergranular attack. These materials therefore were eliminated from consideration. They included Types 304, 347, 310 and 330 stainless steel and Nichrome V.

3. Type 446 was eliminated by scale-adherence test results, the large amount of sigma formed, excessive grain growth, low strength and poor fabricability.

4. The remaining materials can be ranked according to high temperature strength, structural stability and neutron cross section. Type 316 has better high temperature strength than Type 309 and less propensity for sigma formation. It has a lower neutron capture cross section than the nickel base alloys such as Inconel and Inconel X. Among the ferritic materials, Types 405 and 430, DB-2, and FeCrAl exhibited good corrosion resistance but have poor creep strength and are subject to grain growth at 1500 F. DB-2 and FeCrAl have not been fully developed and present fabrication difficulties. Therefore, Type 316 and Inconel should be considered tentatively for high strength, fuel cladding materials.

Acknowledgments

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The writers are indebted to M. T. Simnad for assistance with the theory of corrosion, to L. J. Dijkstra for the x-ray identification of the oxides formed on the surfaces, to G. Schmitt for metallography and to D. Guggisberg, W. H. Ellis and R. J. LaPorte for carrying out many of these tests.

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Any discussion of this article not published above will appear in the December, 1959 issue.

Qualification of Inconel For Nuclear Power Plant Applications*

By H. R. COPSON and W. E. BERRY

Introduction

MATERIALS OF construction for the steam generation system of pressurized water nuclear power plants must pass stringent requirements. The materials are exposed on one side to primary reactor water (heat-exchange medium flowing past the radioactive fuel elements), and on the other side to secondary boiler water.

On the primary side, corrosion products can be transported to the fuel elements, activated, and transported back. This affects the habitability and ease of maintenance. It is imperative, therefore, that there be a minimum of loose corrosion products, and that the induced radioactivity of these products be low. It is also imperative that there be no mixing of the two kinds of water. This requires excellent fabrication characteristics and good corrosion behavior. In addition the materials must have suitable physical and mechanical properties, be commercially available, and reasonable in cost.

Austenitic stainless steels have been used extensively in this application. The corrosion rates are extremely low.[1] From a structural standpoint, the metal loss is insignificant. From the activity standpoint, the stainless steels develop reasonably adherent tarnish films and impart relatively small amounts of corrosion product to the primary water.

Austenitic stainless steels are susceptible, however, to stress corrosion cracking under some conditions.[2, 3, 4, 5] There is some risk that these conditions might be encountered in the heat exchanger systems. Chlorides are likely to be present in the secondary boiler water. Chloride build-up in areas such as crevices in alternately wet vapor phases, plus oxygen and residual or applied stress can promote stress corrosion cracking. Strongly alkaline conditions also can promote cracking.[5, 6, 7]

Inconel* is a material which resists stress corrosion cracking in chloride[3, 8] and alkaline[9] environments. There was interest, therefore, in qualifying this material for nuclear power plant applications. This required radiochemical,[10] welding,[11] and corrosion studies. All of these have been undertaken and all have indicated satisfactory behavior. This paper gives the results of the corrosion studies to date. Other studies are reported elsewhere.[10, 11] The results have been so encouraging that the corrosion tests are being extended to additional water conditions, kinds of specimens and galvanic couples.

★ Submitted for publication April 10, 1959. A paper presented at the 16th Annual Conference, National Association of Corrosion Engineers, Chicago, Illinois, March 16-20, 1959.

* Registered trade mark of The International Nickel Co., Inc.

Experimental Procedure

Outline of Corrosion Tests

Tests were conducted both in simulated primary water and in simulated secondary boiler water. The primary water tests were carried out both in a high velocity loop and in autoclaves.

A normal mill production heat of Inconel was selected for the tests. In addition a heat of unusually high carbon content and a heat of unusually low chromium content were included. These materials were subjected to numerous heat treatments to ensure they could not be damaged in this fashion. Type 347 stainless steel was included as a reference material.

The loop tests were intended mainly to obtain weight loss data under the high velocity conditions which occur in primary water. However, crevice specimens and a few galvanic couples were included in the tests. The autoclave tests were designed to show the effects of crevices, stresses, welds, and of galvanic coupling. Most of the tests were continued for 2000 hours. Specimens were removed periodically for weight change measurements. These indicated both the corrosion rates and the amount of corrosion products lost to the water. At the end of the tests all the remaining specimens were inspected, and additional weight change measurements were made. Microexamination was made of selected specimens in order to detect any selective sub-surface attack.

Water Chemistry

Additions were made to demineralized water of 750,000 ohm-cm specific resistivity to obtain the composition ranges in Table 1. This table gives the results of analyses made during the tests. Fresh water was added at the start of each test period.

The primary water contained 2 to 3 ppm of ammonia. It contained 25 to 65 cc of hydrogen per kilogram except in Tests 4, 5 and 6 where the hydrogen was deliberately omitted. Oxygen content was generally less than 0.02 ppm, except in Test 4 where the water was saturated with oxygen at start-up. The pH was between 8.8 and 9.7. Tests were run at several temperatures between 550 and 680 F. The pressure was 2000 psi at 550 F and 2500 psi at 650 F. The loop tests were opened at 500, 1000, and 2000 hours, and the autoclaves at 200, 500, 1000, and 2000 hours, except for Test 4 which was of 500 hours duration.

The secondary water contained about 50 ppm of chloride added as sodium chloride, and 120 to 150 ppm of phosphate added as disodium phosphate. The pH was adjusted to 10.6 to 10.8 at the start of each period. The solution was saturated with air at start-up and the autoclave headspace contained additional air. Most of the oxygen was consumed when the autoclaves were opened at 200, 500, 1000, and 2000 hours, but the air was re-charged before continuing the tests.

Berry

Copson

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Abstract

In order to qualify Inconel for service in the steam generation system of pressurized water nuclear power plants, the behavior of this material was investigated with respect to resistance to corrosion, radioactivity of corrosion products, and fabrication. The present paper presents the corrosion data. Corrosion tests were conducted in simulated primary and secondary reactor waters on Inconel specimens in several conditions of heat treatment and composition. Stressed specimens, weldments, galvanic couples, and crevice specimens were included. Under all conditions a tightly adherent film formed on the specimens and the corrosion rates were comparable to those of austenitic stainless steels. The Inconel alloy does not suffer intergranular attack or stress corrosion cracking in reactor waters. 6.3.10

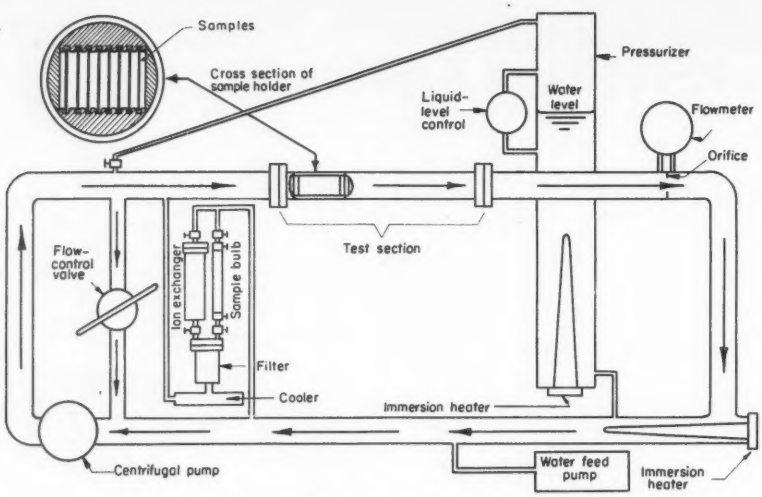


Figure 1—Diagram of high velocity corrosion loop.

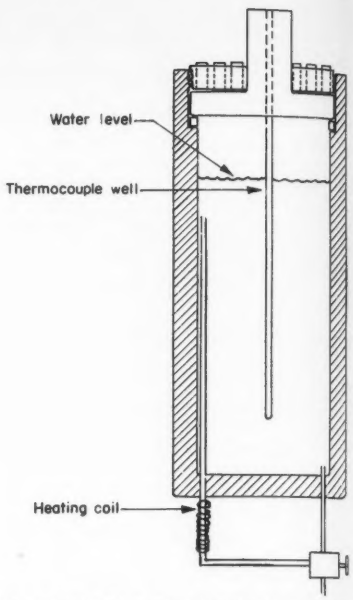


Figure 2—Autoclave adapted with external thermal leg.

Apparatus

Both the high velocity loop and the autoclaves were made of Type 316 stainless steel. A schematic drawing of the loop is shown in Figure 1 and of the autoclaves in Figure 2. In the loop a canned rotor-type of pump gave a velocity of about 30 feet per second past the specimens. The autoclaves were fitted with an external thermal leg to ensure some degree of stirring or circulation of the water during the tests.

Past experience had demonstrated that it was not necessary to electrically insulate the test specimens in any of the tests. Loop specimens were exposed in Inconel holders as shown in Figures 1 and 3. Specimens in the autoclaves were supported from Chromel A hooks.

In the loop tests part of the water was bypassed through a filter to remove suspended crud. A thin sludge, principally Fe_2O_3 from the stainless steel tubing of the loop, was removed from the filters when they were cleaned periodically. Water from the sampling bulbs was always clear, and there was no evidence of suspended matter settling out on standing.

Materials and Heat Treatments

The compositions of the Inconel alloys under consideration are given in Table 2. Alloy 1 represents current production. Alloy 2 was relatively high in carbon content, and Alloy 3 was high in iron and low in chromium. If variations in composition had any harmful effects, it was thought that this selection would reveal them. Type 347 stainless steel quench annealed sheet was used in all the tests as a reference material. Both the stainless steel and the hot rolled mild steel were used in galvanic couples in the autoclave tests.

Alloys 1, 2 and 3 were subjected to

TABLE 1—Analyses of Simulated Reactor Waters

Test	Type of Water	Temperature °F	Exposure Period, Hour	COMPOSITION RANGE pH	NH_3, ppm	H_2, cm^3/kg	O_2, ppm	Cl, ppm	PO_4, ppm
1*	Primary	550	0-500	9.3- 9.5	2.2-3.0	40-65	<0.003-0.002		
			500-1000	9.2- 9.6	2.5-3.1	40-65	0.02 -0.04	.....	.....
			1000-2000	8.9- 9.5	1.4-3.0	40-65	<0.003-0.026	.....	.....
2*	Primary	650	0-500	9.2- 9.3	2.1-3.1	40-60	<0.003	.....	.....
			500-1000	9.1- 9.3	2.1-2.4	40-60	<0.003	.....	.....
			1000-2000	8.9- 9.3	1.9-3.0	40-65	<0.003-0.05	.....	.....
3	Primary	550	0-200	9.1- 9.5	1.7-3.4	26-39	<0.003	.....	.....
			200-500	9.0- 9.2	2.8-3.1	39-48	<0.003-0.04	.....	.....
			500-1000	8.9- 9.6	2.2-3.0	44-54	0.006-0.02	.....	.....
			1000-2000	9.5- 9.6	1.8-3.0	25-52	0.007-0.014	.....	.....
4	Primary	550	0-500	9.2- 9.5	2.6-3.1	.....	0.2 -7.1	.....	.....
5	Primary	550	0-200	9.2	2.7-3.0	< 3	<0.003-0.05	.....	.....
			200-500	9.1	3.0	< 3	<0.003	.....	.....
			500-1000	9.3	2.5-3.0	< 3	<0.003-0.07	.....	.....
			1000-2000	9.3	2.5-3.0	< 3	<0.003	.....	.....
6	Primary	600	0-200	9.1	2.7-3.0	< 3	<0.003-0.05	.....	.....
			200-500	9.3	2.7-3.1	< 3	<0.003-0.007	.....	.....
			500-1000	9.2	2.5-3.0	< 3	<0.003	.....	.....
			1000-2000	9.7	2.4-3.0	< 3	<0.003	.....	.....
7	Primary	680	0-200	8.9- 9.0	2.6-2.9	40-60	<0.003-0.03	.....	.....
			200-500	8.8- 9.2	2.1-2.8	40-60	<0.003-0.05	.....	.....
			500-1000	9.1- 9.2	2.4	40-60	<0.003-0.08	.....	.....
			1000-2000	9.0- 9.1	2.7	40-65	.....	.....	.....
8	Secondary	500	0-200	7.4-10.8	.....	.....	0.2 -7.0	46-50	120-150
			200-500	7.6-10.8	.....	.....	0.2- 7.0	46-50	125-150
			500-1000	10.8-11.3	.....	.....	0.2 -7.0	48-50	129-150
			1000-2000	10.7-11.1	.....	.....	0.1 -7.1	48-50	132-150
9	Secondary	500	0-200	9.8-11.0	.....	.....	0.1 -7.1	49-50	130-133
			200-500	10.9	.....	.....	0.2 -7.0	48-49	134-136
			500-1000	10.8-11.1	.....	.....	0.2 -7.0	50-61	138-139
			1000-2000	10.0-10.8	.....	.....	3.7 -7.1	47-50	130-131

* Loop tests with water velocity about 30 ft./sec. All other tests were in autoclaves.

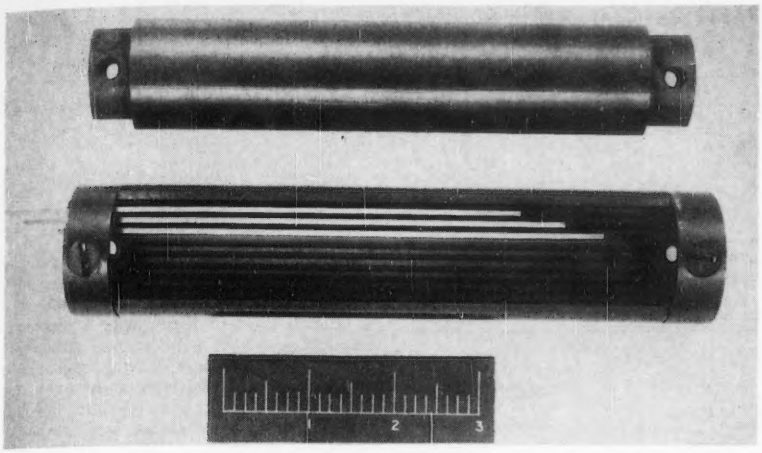


Figure 3—Specimen holder for high velocity loop tests. View with top removed showing three samples partially removed.

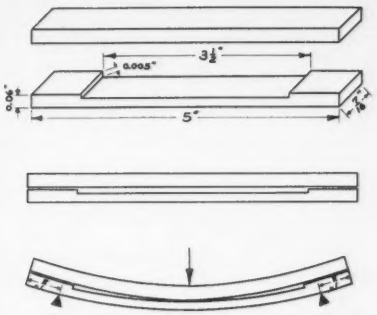


Figure 4—Stress and crevice specimens.

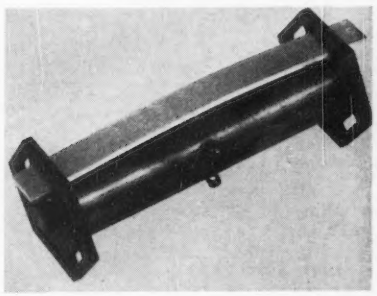


Figure 5—Jig for stress and crevice tests showing one sample deflected.

several different heat treating procedures. The main ones are listed in Table 3. In the table, welding is included as a heat treating procedure. The description of the tests below indicates which heat treated specimens were in which runs. A few specimens were given modifications of the heat treatments listed, but since these specimens behaved like the others, they have been omitted from the discussion.

The object of these heat treatments was to reveal any tendency towards intergranular corrosion, and also to find ways of preventing such attack in case it occurred. In boiling 25 percent nitric acid, mill annealed Inconel, which has been heated subsequently at 1200 F for 2 to 10 hours, suffers intergranular corrosion. On the other hand, prior heating at 1600 F, particularly after cold rolling, decreases the effect of the 1200 F treatment in causing intergranular corrosion in nitric acid. This behavior is analogous to that of stainless steels and is related to carbide precipitation and chromium depletion. Intergranular corrosion of Inconel was not expected in the reactor waters in Table 1. Nevertheless it was considered important to investigate this possibility thoroughly, because there has been one report suggesting such attack.[12]

Types of Specimens

Specimens for the high velocity loop tests were 6 x 0.75 x 0.06 inch in size. They were inserted in the slotted nickel-chromium alloy holders as shown in Figure 3. Crevice specimens were produced by mating two pieces 6 x 0.75 x 0.03 inch in size. These crevice specimens had a 0.125 inch wide slot milled near and parallel to one edge almost the entire length of the specimen. They were mated in such fashion that the slots did not coincide.

In the autoclave tests, stress and crevice specimens were 5 x 0.438 x 0.06 inch in size. They are illustrated by Figures 4 and 5. A step 0.005 inch deep, covering the middle 3.5 inches of the sample, was machined in one-half the samples. A flat sample and a step sample were mated together and stressed in a jig to 90 percent of the room temperature yield strength in the outer fibers of the flat samples. This provided a crevice between the stressed samples ranging from 0.005 inch at the edge of the step to 0 inch at the point of loading.

The remaining specimens for the autoclave tests were prepared from 1 x 2 x 0.06 inch pieces. Standard weight loss specimens were this size. Welds were made by depositing a nickel-chromium alloy filler wire (sold under the trade mark "62") in crisscross fashion on Inconel specimens. Galvanic couples of Inconel to stainless steel and of Inconel to steel were made by placing appropriate specimens end to end and butt welding to give a sample 1 x 4 x 0.06 inch. The inert gas-tungsten electrode process was used with a filler wire such as is sold under the trade mark Inco-Weld "A"*. Additional samples of these same couples were made by bolting appropriate specimens together with an overlap of about one inch. These overlaps, of course, provided a crevice.

Initially all the specimens were ground to give a surface roughness of less than 60 microinches rms. At the start of the tests all the un-welded specimens were cleaned and weighed. At the end of the tests they were re-weighed and then cleaned cathodically, at a current density of 20 amp/dm^2, in hot, inhibited, 2.5 percent sulfuric acid, and weighed again. The cleaning sometimes took as long as 60 minutes, which indicates the adherence of the corosion products. Blank corrections however were nil.

Loop Tests in Primary Water

Two 2000 hour loop tests were run, the first at 550 F and the second at 650 F. Practically identical sets of specimens were included in the tests, the only exception being that a few low-chromium alloy (Alloy 3) specimens with heat treatment 4 were included in Test 1 and not in Test 2. All the other Inconel specimens were of the regular composition with heat treatments 4 and 5. Plain specimens of these types and also of Type 347 stainless steel were exposed for 0 to 500, 0 to 1000, 0 to 2000, 500 to 2000, and 1000 to 2000 hour periods. Crevice specimens were exposed for both 1000 hour periods. In most of the crevice specimens, both parts were the same material, but in a few cases the Alloy 1 composition was mated to Type 347 stainless steel. A few nickel specimens were included in the tests, but the data on these are not reported here.

Autoclave Tests in Primary Water

Five test conditions were used in the autoclave tests in primary water. These are identified as Tests 3 through 7. Table 1 gives the water chemistry and the temperature.

In Test 3 the specimens included: standard weight loss specimens of stainless steel, and of Alloys 1, 2 and 3 with heat treatments 4 and 5, Alloy 2 with heat treatment 1, and similar stress and crevice specimens. Also used were cross welded specimens of Alloys 1, 2 and 3, heat treatment 6, and of Alloy 2, heat treatment 3. Both welded and bolted galvanic couples made using Alloy 1 with heat treatment 4 were used.

In Test 4 exactly the same kinds of specimens were used as in Test 3.

Tests 5 and 6 were confined to standard weight loss specimens of Alloy 1 with heat treatment 4.

In Test 7 the specimens included were standard weight loss specimens of stainless steel, and of Alloy 1 and the high carbon Alloy 2 with heat treatments 1, 2, 7 and 8. Similar Inconel stress and crevice specimens were used. Inconel alloy specimens with cross welds were used also, these specimens being prepared

* Trade mark of The International Nickel Company, Inc.

TABLE 2—Composition of Alloys

Material	Composition, Percent									
	C	Fe	Ni	Cr	Cu	Co	Cb	Mn	Si	S
Alloy 1	0.03	7.52	76.21	15.72	0.14	<0.20	....	0.17	0.18	0.007
Alloy 2	0.08	6.97	76.74	15.67	0.10	0.17	....	0.15	0.26	0.007
Alloy 3	0.04	8.60	76.33	14.44	0.13	0.20	....	0.33	0.10	0.007
347 Stainless	0.05	Bal.	11.0	20.0	....	....	0.70	2.0	0.60	0.024
347 Stainless*	0.06	Bal.	9.3	20.0	....	....	1.20	1.8	1.0	0.022
Steel	0.05	Bal.	0.02	0.01	0.02	....	....	0.47	<0.02	0.021

* 650 F loop test only.

TABLE 3—Main Heat Treatments Applied to Inconel*

Code No.	First Operation	Second Operation	Third Operation	Fourth Operation
1	None	.....	.....	.....
2	1200 F, 10 hr.	.....	.....	.....
3	Welded	.....	.....	.....
4	1600 F, 10 hr.	.....	.....	.....
5	1600 F, 10 hr.	1200 F, 2 hr.	.....	.....
6	1600 F, 10 hr.	Welded	.....	.....
7	Cold Reduced, 40%	1650 F, 0.7 hr.	1200 F, 10 hr.	.....
8	Cold Reduced, 40%	1650 F, 10 hr.	1200 F, 10 hr.	.....
9	Cold Reduced, 40%	1650 F, 0.7 hr.	Welded	1200 F, 10 hr.
10	Cold Reduced, 40%	1650 F, 10 hr.	Welded	1200 F, 10 hr.

NOTE: * All samples started in the mill annealed condition.

according to procedures 9 and 10 in Table 3.

Autoclave Tests in Secondary Boiler Water

In the autoclave tests in secondary boiler water, identical sets of specimens were exposed in the liquid, in the vapor, and at the liquid-vapor interface. Space limitations made it difficult to get as many specimens in the autoclaves as desired. Accordingly in Test 8 many kinds of specimens were included, all of which were exposed for 2000 hours. In Test 9 enough plain weight loss specimens of Inconel were included so that they could be removed after several time periods.

In Test 8 exactly the same kinds of specimens were used as in Test 3 (see above). In Test 9 the only Inconel alloy specimens included were weight loss specimens of Alloy 1 with heat treatment 4. Stainless steel specimens were included in this run, of course, and also some Monel* specimens. The presence of the Monel alloy had no apparent effect on the corrosion behavior of the other materials.

Results and Discussion

Loop Tests in Primary Water

All the specimens in these tests acquired tightly adherent tarnish films. These were dark blue in color except that the stainless steel specimens in Test 1 were almost black. As noted in Table 2, the stainless steel specimens of Test 2 were from a different heat than the others. Covered areas in crevice specimens were slightly lighter in color than areas exposed to flowing water.

The weight measurements indicated that variations in composition and heat treatment had no significant effect. Accordingly the results on the solid specimens have been averaged and are given in Table 4. The weight losses are plotted in Figure 6. Here the maximum and minimum values are shown as well as the average. The standard deviations were less than ± 11 mg/dm^2. It is clear that the initial weight losses were relatively high, and that the rate of attack during the rest of the test was relatively low. After the initial period it is possible to represent the corrosion by a straight line and this has been done in the plots. The corrosion rate corresponding to the slope of this line can be calculated, and the calculated values are given in Figure 6 and in Table 4.

The main effect of raising the temperature from 550 to 650 F was one of changing the amount of initial attack. This was increased in the case of Inconel and decreased in the case of stainless steel. However, as already noted, two lots of stainless steel were used in these tests and this may explain this result. From 500 hours on there was not too much difference in the slopes of the lines, which seems to indicate that all the corrosion products were about equally protective. It should be noted that all the corrosion rates were exceedingly low. A corrosion rate of 10 mg/(dm^2) (month) is about equivalent to 0.000055 inch penetration per year.

X-ray and electron diffraction techniques showed that the corrosion films on Inconel consisted of nickel oxide (NiO) and spinel ($NiCr_2O_4$), that those on stainless steel consisted of spinel, and those on steel (in the autoclave tests) consisted of magnetite (Fe_3O_4). X-ray fluorescent analyses revealed about the same ratio of metal atoms in the corrosion product as in the parent metal.

This information makes it possible to calculate approximately the total weight of corrosion products from the descaled metal loss. The weight measurements gave the portion of the corrosion products adhering to the specimens (Table 4). The difference is the amount of corrosion products lost to the flowing water streams. These values are plotted in Figure 7. The results roughly parallel the metal loss plots in Figure 6. The rate of release of corrosion products was relatively high at the start of the tests and then quickly dropped to low values.

In the case of the crevice specimens, weight losses in general ran somewhat lower than for non-crevice specimens. Apparently, the protected areas were less affected by the action of the flowing water. Coupling Inconel and stainless steel in the crevice specimens had no effect on the amount of corrosion of each metal.

Metallographic examination of selected specimens revealed no intergranular attack, no pitting, and no selective attack of any type in the Inconel alloy specimens. The stainless steel specimens exposed to the 650 F water had some extremely shallow pits, less than 0.5 mil deep at 2000 hours. Otherwise the stainless steel showed no selective attack.

Autoclave Tests in Primary Water

The weight loss data for the plain specimens in these autoclave tests were plotted against time in the same manner as for the loop tests. As before the initial weight losses were relatively high, but from then on a straight line could be drawn through the points. Figure 8 summarizes the results obtained at 550 F in Test 3. All the Inconel alloy data grouped closely together except for curve B, which pertains to some mill annealed specimens of the high carbon Alloy 2. As soon as this was realized, some twelve additional mill annealed specimens (four of each of the Alloys 1, 2 and 3) were inserted in the autoclaves at the time of the 500 hour opening. Weight losses were obtained on these specimens after 1500 hours of exposure and averaged 12.2 mg/dm^2, with very little scatter. This is close to the other curves, so that curve B seems out of line for reasons unknown. Mill annealed specimens behaved normally in all the other tests. It is concluded that variations in composition and heat treatment of the nickel-chromium alloys had no significant effect.

The average corrosion rates of stainless steel and Inconel from 200 hours on can be calculated from the slopes of the lines in Figure 8. These were 5.4 and 1.5 mg/(dm^2) (month), respectively. These were less than in the high velocity loop tests, as would be expected.

In Test 4 the hydrogen was omitted and the water was saturated with oxygen at start up. The average weight losses after 500 hours are given in Table 4. These did not differ greatly from the results of Test 3 (Table 4 and Figure 8). The presence of some oxygen in the water was not detrimental to Inconel or stainless steel.

* Monel is a registered trade mark of The International Nickel Co., Inc.

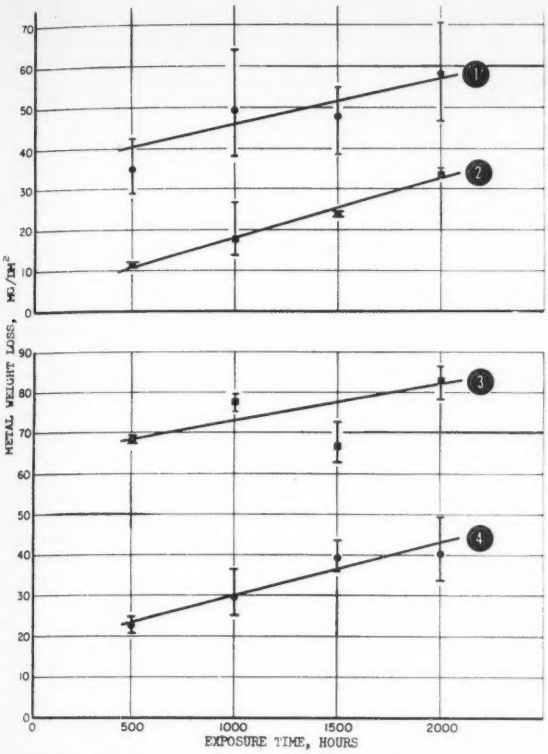


Figure 6—Weight losses in flowing primary water, Tests 1 and 2. Code to numbers is as follows: (1) Inconel: 7.6 mg/dm² per month; (2) Stainless steel: 10.1 mg/dm² per month; (3) Stainless steel: 7.4 mg/dm² per month; (4) Inconel: 8.8 mg/dm² per month. Top two graphs are for 650 F and bottom two graphs are for 550 F.

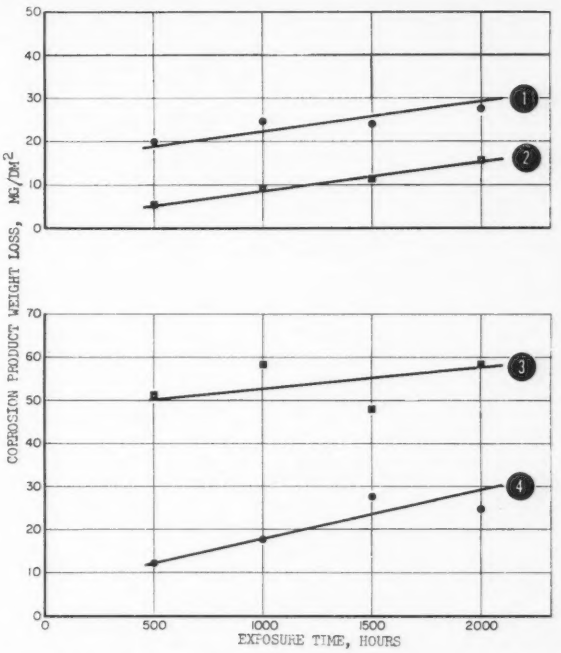
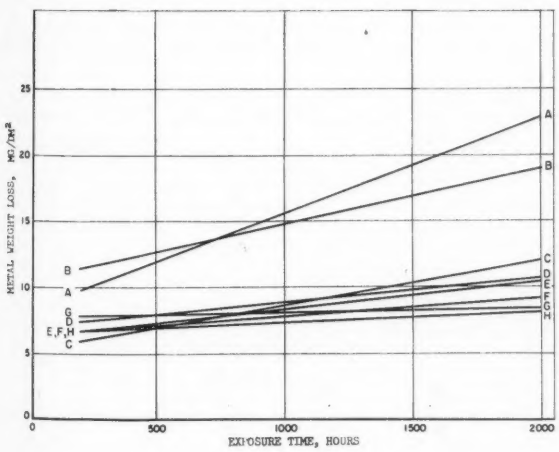
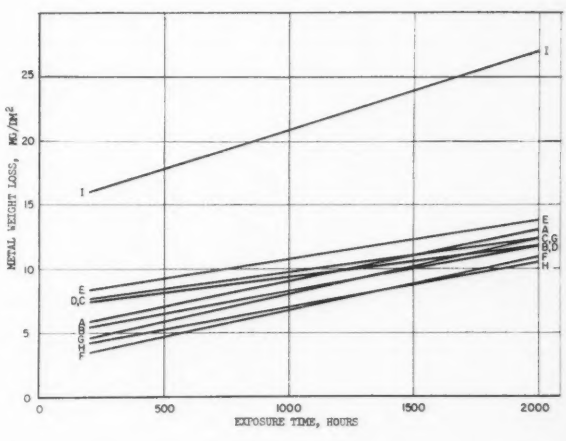


Figure 7—Weight of corrosion products lost to flowing primary water, Tests 1 and 2. Code to numbers is as follows: (1) Inconel: 4.7 mg/dm² per month; (2) Stainless steel: 4.9 mg/dm² per month; (3) Stainless steel: 3.7 mg/dm² per month; (4) Inconel: 8.0 mg/dm² per month. Top two graphs are for 650 F and bottom two graphs are for 550 F.



MATERIAL	Heat Treatment	Rate, mg/(dm²) (month)
D. Alloy 1	4	1.3
H. Alloy 1	5	0.6
B. Alloy 2	1	3.1
G. Alloy 2	4	0.2
C. Alloy 2	5	2.5
E. Alloy 3	4	1.5
F. Alloy 3	5	1.0
A. Stainless steel	..	5.4

Figure 8—Weight losses in static 550 F primary water, Test 3.



MATERIAL	Heat Treatment	Rate, mg/(dm²) (month)
A. Alloy 1	1	3.3
B. Alloy 1	2	2.9
C. Alloy 1	7	2.4
D. Alloy 1	8	2.1
E. Alloy 2	1	2.6
F. Alloy 2	2	3.0
G. Alloy 2	7	3.1
H. Alloy 2	8	2.6
I. Stainless steel	..	4.4

Figure 9—Weight losses in static 680 F primary water, Test 7.

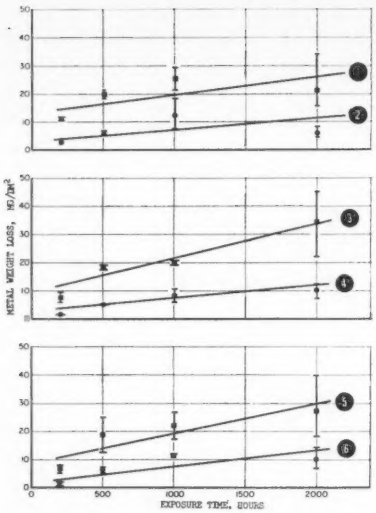


Figure 10—Weight losses in static 500 F secondary water, Test 9. Code to letters is as follows: (1) Stainless steel: 5.0 mg/dm² per month; (2) Inconel: 3.2 mg/dm² per month; (3) Stainless steel: 8.9 mg/dm² per month; (4) Inconel: 3.9 mg/dm² per month; (5) Stainless steel: 7.9 mg/dm² per month; (6) Inconel: 3.9 mg/dm² per month. Top two graphs are for vapor, middle two are for interface and bottom two are for liquid.

In Tests 5 and 6 the effect of not adding hydrogen to the water was studied further. These tests differed from Test 4 in that the oxygen content of the water was low. The weight loss results are included in Table 4. The average corrosion rates obtained from the flat portions of the weight loss versus time curves were slightly lower than in Test 3. Thus, a high hydrogen content in the primary water does not appear necessary to ensure the excellent corrosion resistance of Inconel. In Test 6 the temperature was 600 F, instead of the 550 F used in Tests 3, 4 and 5. This change had no marked effect.

In Test 7 the temperature was raised further to 680 F, but otherwise the water chemistry was the same as in Test 3. Figure 9 summarizes the results on the weight loss specimens. The similarity to Figure 8 is striking. The average slope for the Inconel specimens was increased slightly, but the average corrosion rate continued low, 2.7 mg/(dm²) (month). Heat treatment and composition continued to have negligible effect, even though some severe heat treatments were included in this test.

Weight losses also were obtained on the stress and crevice specimens and on the bolted galvanic couples. These ran about the same as on the fully exposed specimens.

As in the loop tests, the Inconel and stainless steel specimens acquired thin adherent tarnish films. These were straw to purple in color. Fairly heavy black films formed on the steel samples. In Test 4 with air present some areas of the steel showed red Fe_2O_3.

Careful inspection was made of the specimens. In the case of the Inconel and stainless steel specimens there was no evidence of pitting or intergranular attack in crevices, in stressed areas, near welds, or anywhere else. This was confirmed by numerous metallographic examinations. The steel specimens suffered from some shallow randomy located pits which were not associated with crevices or galvanic effects. The galvanic couples seemed to have no effect on either member of the couples.

Autoclave Tests in Secondary Boiler Water

Specimens exposed to boiler water acquired adherent tarnish films, just as in the other tests. These were tan to straw colored on Inconel. They were very light colored in the vapor phase and darker totally immersed. The tarnish films on the stainless steel were darker than on Inconel. The steel specimens developed fairly heavy black films.

Weight loss measurements again showed that variations in the composition and heat treatment of the Inconel had negligible effect. The results on the flat 2 x 1 inch specimens are summarized in Table 5. The data from Test 9 are plotted in Figure 10. As in the other tests the corrosion was relatively rapid during the first exposure period, and from then on the corrosion could be represented by a straight line. The calculated corrosion rates corresponding to the slopes of the lines are given in Figure 10 and Table 5. Inconel corroded somewhat less rapidly in the vapor than when totally immersed or exposed at the liquid line and corroded less rapidly than the stainless steel. The corrosion rates were a little higher than in the static primary water. However, all the corrosion rates were again exceedingly low.

Inspection of the Inconel specimens again showed no pitting or local attack. The stresses, crevices, welds, and galvanic couples had no significant effect on this material. Weight losses on the stress and crevice specimens and on the bolted couples ran no higher than on the fully exposed specimens. Metallographic examination of numerous cross sections again confirmed the absence of any intergranular or selective attack.

There was no indication that Inconel accelerated the corrosion of the other members of the galvanic couples. This was particularly true in the case of stainless steel. On the welded couples the steel showed some pitting, but the pitting was not concentrated near the welds. On the bolted couples the steel was pitted

TABLE 4—Corrosion Results in Primary Water

Test	Exposure Time, Hr	No. of Specimens		Weight of Corrosion Products on Specimens, mg/dm²		Wt. Loss, mg/dm²		Avg. Corrosion Rate After First Period, mg/(dm²) (month)*	
		Inconel	347	Inconel	347	Inconel	347	Inconel	347
1	500	14	3	16.9	47.6	22.3	68.4		
	1000	12	2	20.4	52.3	29.5	77.4		
	1500	14	3	23.5	48.4	39.3	66.7		
	2000	12	3	27.2	61.5	40.3	82.9	8.8	7.4
2	500	14	3	26.2	10.3	35.0	11.4		
	1000	12	3	40.1	15.6	49.2	17.8		
	1500	14	3	38.8	21.7	47.9	23.9		
	2000	12	3	47.9	31.0	58.1	33.8	7.6	10.1
3	200	18	4	7.8	10.7	7.7	8.8		
	500	18	4	7.9	19.7	8.1	16.0		
	1000	18	4	8.9	20.0	8.3	15.5		
	2000	36	8	12.1	28.7	10.8	20.7	1.5	5.4
4	500	32	4	14.1	13.7	9.2	12.7		
5	200	8		10.5		10.8			
	500	8		9.4		8.3			
	1000	8		15.9		11.1			
	2000	8		10.2		10.7		1.0	
6	200	8		9.8		7.7			
	500	8		9.4		6.7			
	1000	8		14.9		9.5			
	2000	8		15.7		10.0		1.1	
7	200	16	2	8.0	20.1	6.1	18.0		
	400	4		7.2		4.7			
	500	12	2	9.8	20.5	6.2	16.8		
	700	4		10.6		5.1			
	1000	12	2	9.5	20.6	7.7	18.1		
	1200	4		10.3		8.4			
	2000	12	2	17.8	35.1	12.6	28.7	2.7	4.4
	2200	4		20.1		12.1			

* Calculated from slope of best straight line through plotted points.

TABLE 5—Corrosion Results in Secondary Boiler Water

Test	Material	Exposure Time Hr.	No. of Specimens	Wt. of Corrosion Products on Specimens, mg/dm²			Wt. Loss mg/dm²			Avg. Corrosion Rate After First Period mg/(dm²) (month)*		
				Vapor	Interface	Liquid	Vapor	Interface	Liquid	Vapor	Interface	Liquid
8.....	Inconel	2000	28	4.9	15.5	12.7	5.8	11.1	10.0			
8.....	347	2000	4	32.9	22.1	15.0	28.3	22.3	18.7			
9.....	Inconel	200	2	7.1	5.5	4.8	3.0	1.8	1.6			
	Inconel	500	2	8.1	7.5	9.7	6.0	5.0	6.2			
	Inconel	1000	2	15.2	15.2	19.4	12.9	8.5	11.5			
	Inconel	2000	4	14.1	19.6	18.3	6.2	10.4	10.5	3.2	3.9	3.9
9.....	347	200	2	19.5	12.8	10.0	11.2	7.8	6.7			
	347	500	2	27.0	23.0	22.7	20.0	18.6	18.8			
	347	1000	2	38.1	28.0	25.3	25.6	20.0	22.1			
	347	2000	4	33.3	42.0	30.1	21.5	34.7	27.6	5.0	8.9	7.

* Calculated from slope of best straight line through plotted points.

severely in the crevice area covered by Inconel. This attack is believed to have been caused by the crevice rather than by the galvanic couples.

There was no stress corrosion cracking on any of the stressed specimens, either of the Inconel or stainless steel. Although 50 ppm of chloride ion was present in the secondary water, the test conditions evidently did not allow local build-up or concentration of the chloride ion, such as is usually associated with stress corrosion cracking of stainless steel.

Conclusions

The results of this study indicate that, from a corrosion standpoint, Inconel is an excellent construction material for primary and secondary waters of pressurized water reactors.

Inconel forms tightly adherent tarnish films in the waters studied. It contributes a relatively small amount of corrosion product to a high velocity stream of primary water. It corrodes at rates comparable with those of Type 347 stainless steel.

Variations in composition and heat treatment have little effect on the corrosion behavior of Inconel in primary and secondary waters. Applied stress, crevices, or welding do not cause selective or intergranular attack. Galvanic coupling of Inconel to steel or to stainless steel does not accelerate the attack on either member of these couples.

Acknowledgment

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Any discussion of this article not published above will appear in the December, 1960 issue.

Effects of Velocity on Corrosion*

By H. R. COPSON*

Abstract

Discusses effects of velocity of fluids on corrosion rate of metals. Explains relation between corrosion rate factors and velocity and velocity effect on corrosion of iron and other metals. Also discusses galvanic effects produced by changes in velocity. Includes discussion of erosion corrosion of iron, copper alloy and other alloys plus explanation of cavitation corrosion. 3.5.11

Introduction

WITHOUT MOVEMENT of some kind there would not be any corrosion. For corrosion to occur material must be brought into contact with the metal surface and the metal atoms must move away. Therefore the rate of motion of the environment with respect to a metal surface is a major factor in the corrosion of metals. The effects of velocity, however, are many and varied. There is no simple answer to the question of what an increase in velocity will do. It is necessary to know the details of the exposure conditions and also what metals are involved.[1]

General Effects of Velocity

In supposedly stagnant solutions, conditions are not necessarily uniform. A little motion often tends to make the environment more uniform and thereby tends to make corrosion more uniform and to prevent local attack. On the other hand, more violent motion may set up turbulence, possibly producing non-uniform conditions and causing pitting. Thus the effect of increased velocity on pitting and local attack will be different in different cases.

Motion generally increases total weight loss by supplying the corrosives at a faster rate. Motion thins quiescent layers at the metal surface so that corrosives can more easily reach the metal. At high velocity, character of the corrosion products may change and become more protective. Motion can decrease corrosion by replenishing inhibitors of corrosion, particularly at crevices or other dead places. Thus the general rule is that corrosion increases with velocity, but the effect can be just the opposite in special cases.

Different metals and alloys are affected differently by changes in velocity. Velocity can have a marked effect on galvanic behavior when metals are coupled together. Measurements at different velocities can lead to conflicting conclusions with regard to galvanic corrosion.

When the velocity becomes high, mechanical effects add to the damage to metals. Erosion can completely remove protective films or layers of corrosion products and keep corrosion going at a high rate. Damage caused by cavitation-erosion is related to velocity effects.

Relation Between Corrosion Rate Factors and Velocity

The velocity itself should be known, but true velocity is difficult to measure. Friction films travel with moving specimens and adhere to stationary ones. Also velocity is apt to vary from point to point. Even at supposed zero velocity, convection currents and streaming effects of corrosion products moving under the influence of gravity make the true velocity uncertain. Accordingly, the effects of velocity should be studied under more than one set of conditions.

Oxygen concentration is often a major factor in controlling rate of corrosion. In most cases increased velocity would be expected to increase the total corrosion by suppling more oxygen. However, oxygen may not be distributed uniformly. Local variations in oxygen content can set up concentration cells and cause pitting. Such variations might be caused by deposits on the surface, in which case an increase in velocity might remove the deposits and eliminate pitting. But differences in velocity from point to point can set up severe differential aeration cells.

For an example, a metal covered with a rather poor oxide film can be considered. At unaerated places where the film is highly discontinuous, the potential would approach that of the film-free metal. At aerated places, aeration would keep the film in better repair, and the potential would rise towards that of solid oxide. Aeration would make the open circuit potential of cathode areas even more cathodic. Thus the strength of an oxygen concentration cell is increased by an increase in velocity at the cathode.[2]

Another type concentration cell affected by motion is a metal ion cell. Where the velocity is highest, the metal ions formed by corrosion will be swept away more effectively, and such areas will tend to become anodic. The oxygen concentration cell and the metal ion cell oppose each other; both increase in intensity with velocity. With noble metals such as copper, removal of metal ions can predominate in determining corrosion behavior. With less noble metals such as iron, the oxygen concentration cell usually predominates. The principal determining factors are differences in oxygen concentration and velocity. As the differences in oxygen concentration increase, the velocity of the liquid required to cause reversal from an oxygen cell to a metal ion cell also increases.[3]

Corrosion product films are another major factor in determining corrosion rates. Because velocity affects the condi-

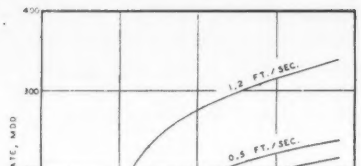

Figure 1—Effect of velocity and oxygen concentration on corrosion of steel.[8] Five to seven-day tests in 23 C water.

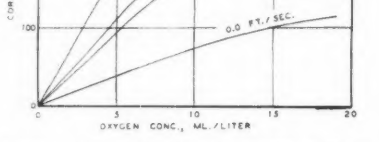

Figure 2—Effect of velocity on corrosion of rotating steel cylinders. One-day test in oxygenated water.[9]

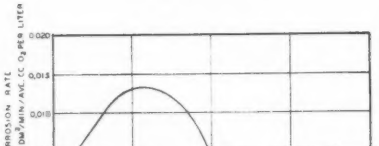

Figure 3—Effect of velocity on corrosion of ½-inch pipe in oxygenated water.[10]

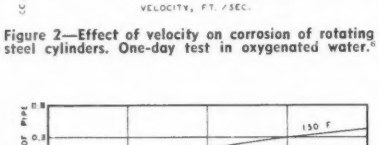

Figure 4—Effect of sea water velocity on corrosion of steel at atmospheric temperature, exposed 38 days.[13]

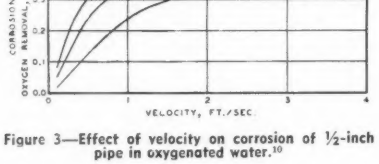

Figure 5—Effect of velocity on potential of steel and copper in sea water.[16]

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* Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

tions under which the films are formed, it can affect their protective character. For example, at high velocity, due to the greater availability of oxygen, ferric hydroxide might be precipitated in close contact with a metal surface and be protective, whereas at some lower velocity ferrous products might be precipitated away from the metal and be nonprotective.

Corrosion rates usually decrease with time. This is due in part to gradual protective film thickening. If the films are thick enough, velocity changes will be unnoticed at the metal surface. At high velocity, however, erosive action can remove the protective films. Impingement attack can cause this to occur locally and lead to rapid pitting.

Composition of the solution is another important factor. Corrosives present in small concentration might soon be depleted unless some flow is present to have a replenishing action. Similarly, if an inhibitor of corrosion is present, some flow is necessary to distribute the inhibitor.

Temperature and pH should not be neglected as corrosion rate factors. However, there is little connection between these and velocity except as the other factors discussed come into play. Differential aeration cells are less potent at low pH than at high pH.

Velocity Effect on Iron Corrosion

Corrosion behavior of iron illustrates many of the principles which have been discussed.

Velocity and Passivity

Figure 1 shows data obtained in tap water at Cambridge, Mass.[3] The speed of rotation of steel specimens was varied and also the oxygen content of the water. Corrosion increased with velocity and oxygen concentration. Tap water saturated with air at room temperature contained about 6 ml/liter of oxygen. At this concentration the corrosion rate increased from 47 mdd at zero velocity to 208 mdd at 1.2 ft/sec.

The maximum oxygen concentration shown in Figure 1 is about 18 ml/liter. At higher oxygen concentrations such as 24 ml/liter, the iron would be expected to acquire a protective oxide film.[4, 5] Passivity would set in and the corrosion rates would be low. However, the effects of velocity and oxygen concentration are inter-related. A higher velocity would bring sufficient oxygen to the iron surface so that passivity can set in at oxygen concentrations of 6 ml/liter or less.[6-9]

This is illustrated by Figure 2. These data also were obtained by rotating steel cylinders in Cambridge tap water.[6] The corrosion rate increased with velocity to about 1.3 ft/sec., and then decreased with higher velocities to a minimum of about 2.3 ft/sec. With still higher velocities the protective film would be eroded away and corrosion would increase again.

As might be expected, position of the maximum and minimum will vary with conditions. Cold water dissolves more oxygen than hot water and thus favors the development of passivity. Turbulence and entrained air bubbles also favor getting the oxygen to the metal surface. When cold water was run through a beaker containing iron specimens in another experiment, the maximum was observed at speeds as low as 0.004 ft/sec.[7]

For passivity to set in, the surface must be clean.[8] With rusted specimens, corrosion at high velocity is at a rapid rate. Undoubtedly the rust prevents oxygen from reaching the corroding surface. Hence it is not safe to place much reliance on iron becoming passive at high velocity.

This is illustrated by data in Figure 3, which were obtained by flowing water through pipe.[10] The corrosion rate increased with velocity, sometimes approaching a maximum but never decreasing. In some tests the velocity was as high as 8 ft/sec. Presumably the pipe did not have a clean surface at the start. Other tests have shown that, even when passivity does not set in at high velocity, it will not persist after the motion has stopped.[11]

A passive condition is not apt to develop if the water contains large amounts of chlorides.[12] Some data obtained in sea water are shown in Figure 4. Here the corrosion rate increases with velocity to 25 ft/sec at least. High concentrations of dissolved oxygen have failed to produce passivity of steel in salt solutions.[5]

Differential Aeration Cell

Even though the entire surface may not go passive, large areas may show negligible corrosion due to development of differential aeration cells. Areas at high velocity are aerated better and develop a more noble potential than areas at lower velocity. The effect of velocity on the potential of steel in sea water is shown in Figure 5. As the velocity increased from 0 to 14 ft/sec, the steel became about 130 mv more noble. Tap water in Ottawa, Ontario, had potential differences as high as 700 mv.[9]

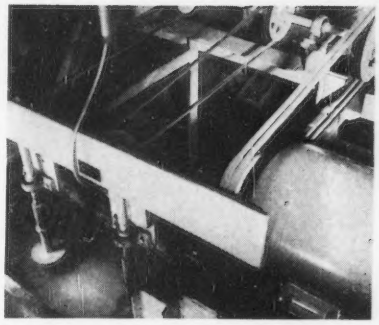

Figure 6—Apparatus to rotate specimen discs in sea water.[15]

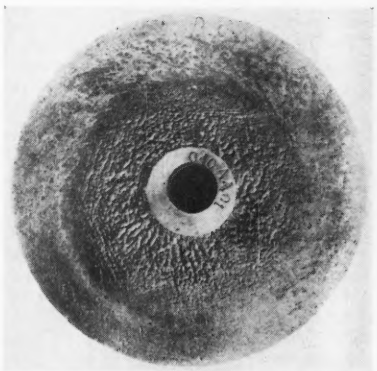

Figure 7—Corrosion on iron disc (5-inch diameter) rotated at 1140 rpm for 30 days in sea water.[1]

Effect of a differential aeration cell on corrosion of iron can be shown by rotating discs of metal in sea water. Apparatus for this experiment is shown in Figure 6. The disc was rotated horizontally while totally immersed in quiet sea water. The result on a cast iron disc is shown in Figure 7. This disc (5 inches in diameter) was rotated at 1140 rpm for 30 days. Velocity at the outer edge was 25 ft/sec. Average corrosion rate was 100 mdd, but deep attack was con-

TABLE 1—Results of Metal Exposed to Quiet and Moving Salt Water at Freeport, Texas[37]

Material	CORROSION RATES MDD		PITTING, MAX., INCHES	
	Quiet Water	Moving Water*	Quiet Water	Moving Water*
Admiralty Brass	2	24	None	Severly eroded from upstream side.
Type 316 Stainless Steel	1	0	0.062	None

* Moving water velocity = 4 ft/sec.

TABLE 2—Effect of Velocity on Galvanic Corrosion of Steel in Sea Water[1, 37]

Temperature ..... 50 F
Time ..... 16 to 19 days
Areas ..... 0.2 sq dm

Couple	VELOCITY 0.5 FT/SEC		VELOCITY 7.8 FT/SEC	
	Corrosion Rate of Steel mdd	Galvanic Effect, mdd	Corrosion Rate of Steel, mdd	Galvanic Effect, mdd
Steel to Steel	60*	..	170*	..
Steel to 18-8 Stainless	141	81	195	25
Steel to Titanium	139	79	224	54
Steel to Copper	119	59	525	355
Steel to Nickel	117	57	607	437

* Average of both members of couple.

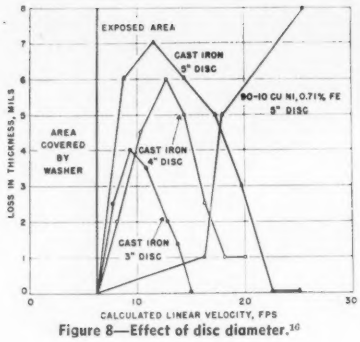
EXPOSED AREA
CAST IRON 5" DISC
90-10 CU NI, 0.71% FE 5" DISC
AREA COVERED BY WASHER
CAST IRON 4" DISC
CAST IRON 3" DISC
LOSS IN THICKNESS, MILS
CALCULATED LINEAR VELOCITY, FPS

Figure 8—Effect of disc diameter.[16]

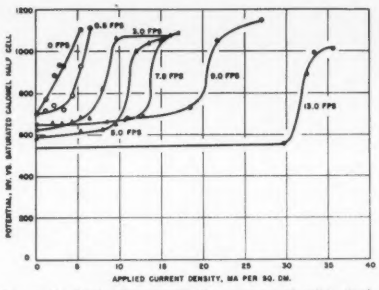

Figure 9—Cathodic polarization curves of mild steel in sea water flowing at different velocities.[14]

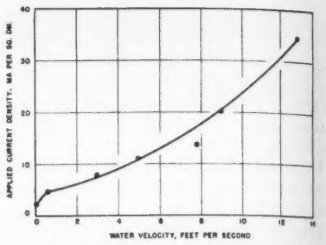

Figure 10—Effect of velocity on current required to polarize steel to a potential of 800 mv.[14]

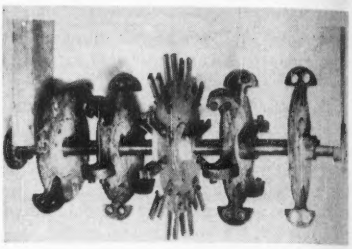

Figure 11—Test specimens mounted on periphery of large discs in a test for erosion corrosion.[20]

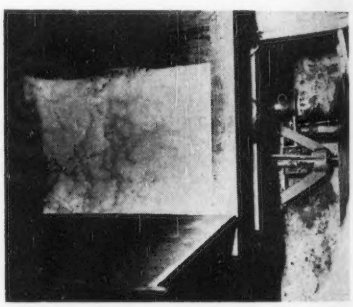

Figure 12—Apparatus used to rotate discs in vertical plane so that air is sucked into the solution to produce a thick froth, seen at left.[21]

fined to the central portion. Outer portion of the disc which was moving at high velocity showed no attack.

That this result is due to a differential aeration cell can be confirmed by rotating discs of smaller diameter. A disc 3 inches in diameter would just cover the deeply corroded area in the figure. If no current flowed between the inner and outer portions of the disc, this 3-inch disc should corrode like the inner portion.

Figure 8 summarizes results obtained on cast iron discs 3, 4, and 5 inches in diameter.[16] Loss in thickness is plotted against the calculated linear velocity in ft/sec, which corresponds to the distance from the center of the disc. The 3-inch disc showed the same pattern of attack as the 5-inch disc with the outer portion showing negligible attack and the deep attack being confined to the inner portion. It is significant that attack depth increased with disc size. The larger the disc the greater the differences in velocity and the greater the strength of the differential aeration cell.

Effect of Velocity on Corrosion of Other Metals

In Figure 5, a curve is included showing the effect of velocity on the potential of copper in sea water. The potential of copper becomes less noble as the velocity increases, probably due to removal of copper ions. In any case, velocity affects iron and copper differently.

A curve is included in Figure 8 showing the distribution of attack on a rotating disc of a high copper alloy. The pattern is entirely different from that on cast iron. Here the deepest attack occurred on the areas of highest velocity. Undoubtedly there was current flow between the different portions of the disc with, in this case, the areas of highest velocity being anodic.

Each metal or alloy should be considered separately. This is emphasized by data in Table 1, which gives results of 42-month immersion tests in salt water at Freeport, Texas.[17] The brass was more vulnerable to attack in flowing sea water than in quiet sea water. Stainless steel was substantially free from attack in flowing sea water but suffered severe pitting under fouling organisms in quiet water.

This stainless steel behavior is of considerable practical importance. In a heat exchanger, it would be preferable to have sea water flow through the tubes rather than through the shell around the tubes. Comparatively stagnant areas outside the tubes could cause severe pitting. Inside the tubes the desired velocity of flow is easier to control.[18]

Galvanic Corrosion

Due to difference in behavior of different metals, changes in velocity can produce surprising galvanic effects. Some examples are given in Table 2, which gives the corrosion rate of steel when coupled to another piece of steel or to 18-8 stainless steel, titanium, copper or nickel in sea water.[1,17] At a velocity of 0.5 ft/sec, corrosion rate of steel is 60 mdd. It is increased by coupling with any of the metals mentioned, and the galvanic acceleration of corrosion of steel is approximately the same in all cases, running 57 to 81 mdd. At a velocity of 7.8 ft/sec, the corrosion rate of steel is 170 mdd. This is increased only slightly by coupling with 18-8 stainless steel or with titanium but is increased greatly by coupling with copper or nickel.

The answer lies in the cathodic polarization curves.[1,17,19] The increased velocity increases oxygen supply to the cathodes. This makes it more difficult to polarize the copper and nickel cathodes and increases the galvanic current supplied by these couples. On the other hand, increased oxygen supply makes the protective oxide films on the stainless steel and titanium even more perfect. As a result, these cathodes polarize more readily at high velocity than at low velocity. Stainless steel and titanium supply less galvanic current at high velocity than at low velocity.

Cathodic polarization behavior of iron becomes important when attempts are made to prevent corrosion by cathodic protection. Figure 9 shows the cathodic polarization curves of mild steel in sea water flowing at different velocities. As the velocity increases, the curves shift to the right. At high velocity, steel is much more difficult to polarize.

The current necessary to polarize steel to a potential of 800 mv can be read from the plot for each velocity. Figure 10 shows how this current increases with velocity. Relatively little current is required at zero velocity. At high velocity much more current is required to cathodically protect steel. If zinc or some other material is used as a sacrificial anode, the anode must supply more current and will be consumed faster under conditions of high velocity.

Erosion Corrosion of Iron

As the velocity increases, erosion starts to strip away protective films.[6,8] In the case of iron, this action is opposed by the increased oxygen supply which tends to keep such films in repair. If a differential aeration cell has become established, it will protect areas exposed to high velocity. However, in the absence of a strong aeration cell, it is possible for the erosive action to be severe. Areas stripped of their film then become anodic to adjacent filmed over areas.[20]

One test method of achieving this effect is shown in Figures 11 and 12. Test specimens were mounted on the periphery of large discs. Some test specimens were flat bars; others were short sections of tubes. These discs were rotated in a vertical direction, thus sucking air into the solution and producing a thick froth as shown in Figure 12. This frothing action supplied air equally to all parts of the specimen so that differential aeration cells were not set up. As a result protective films can be eroded away.

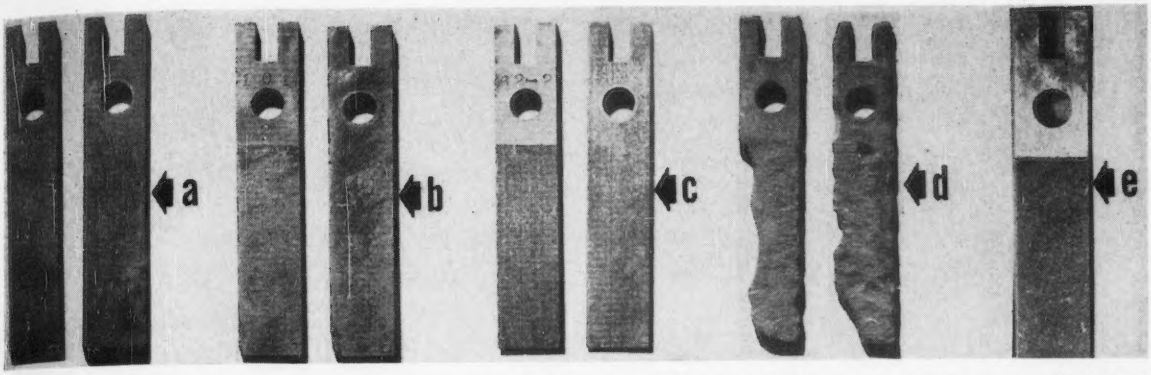

Figure 13—Appearance and weight loss of specimens rotated in sea water at top speed of 27 fps. Specimen (a): Ni-Resist Type 1; 1.03 g loss. Specimen (b): Ni-Resist Type 2; 4.45 g loss. Specimen (c): Ni-Resist Type 3; 0.93 g loss. Specimen (d): cast iron; 21.79 g loss. Specimen (e): Composition G bronze; 5.79 g loss.[22]

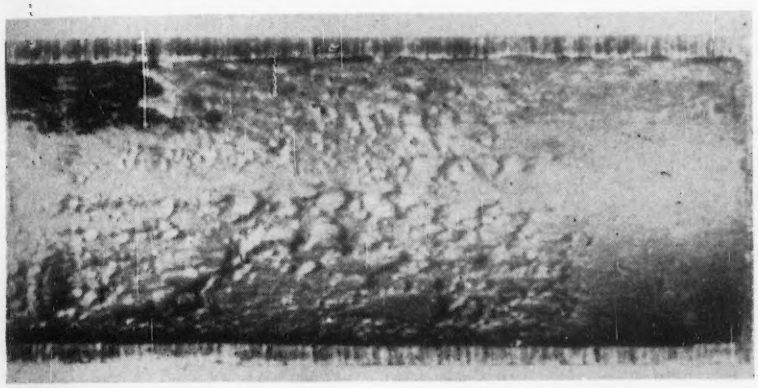

Figure 14—Impingement attack of low-iron 70-30 cupro-nickel tube in sea water.[21]

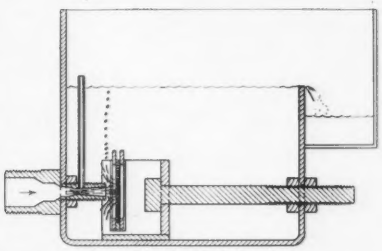

Figure 15—Apparatus for aspirator type jet test.[21]

Figure 13d shows cast iron specimens after 60 days in sea water in this test. Velocity at the tip of the specimens was 27 ft/sec. Weight loss was equivalent to 878 mdd but was confined mostly to the leading edge. This type attack is called erosion corrosion and obviously can be severe. The damage, however, is caused by corrosion. Specimens of inert materials such as gold would not show attack under these conditions even though they were softer and less resistant to erosion than iron.[23]

Figure 13 also shows the results on other cast materials. Ni-Resist and bronze specimens were substantially unattacked in these tests. The Ni-Resists are austenitic nickel alloy cast irons. These results show that alloy materials which develop better and more adherent films are more resistant to erosion corrosion. In general, anything in the test conditions which affects the character of protective films will have some effect on resistance to erosion corrosion.[24]

Erosion Corrosion of Copper Alloys

Erosion corrosion of copper alloys often is called impingement attack, occurring where a turbulent aerated water stream impinges against a metal surface.[25] An example is shown in Figure 14, showing the appearance of a tube specimen of low-iron 70-30 cupro-nickel after exposure in the apparatus of Figures 11 and 12. Turbulent water entering the tube caused a continuous local breakdown and removal of protective films from the metal surface by impingement. With removal of protective films, the metal was anodic to adjacent areas and suffered rapid corrosion. Impinging aerated water depolarizes cathode areas adjacent to the anodes so that ideal conditions for rapid electro-chemical corrosion are maintained.

Much can be accomplished by better design to avoid local high velocities and turbulence in circulating waters. However, impingement attack generally can be avoided by use of resistant alloys which form good protective films.[26] A jet impingement test is often used[27,28] to compare alloys.

Figure 15 shows one form of a jet impingement test. The apparatus causes a high velocity water stream to impinge on the surface of a test specimen held in front of the submerged jet. Shape of the jet is such that air can be sucked into the jet stream through a tube projecting above the water.

Sea water or salt solution generally is used for these tests because it is more corrosive than plain water. In fresh sea water, slimes and minute shells from marine organisms may increase the attack, as sand would do. In pure filtered or re-circulated sea water, presence of air bubbles increases the attack. Size of the area exposed to the jet compared with the rest of the specimen also can influence attack depth. As in any test, it is important to correlate the results with service performance. For this purpose, it may be desirable to vary the severity of test conditions.

Type of attack produced by the jet tests is shown in Figure 16. Corrosion is severe where the jet impinges and the protective film is eroded away. The surrounding area shows little corrosion. This specimen was a low-iron 70-30, cupro-nickel, exposed to a jet of sea water containing entrained air bubbles. Peak velocity was 12 ft/sec. The type attack is similar to that in the tube in Figure 14.

Figure 17 shows the behavior in the jet test of an alloy highly resistant to impingement attack. Conditions were similar to those described for Figure 16. The only difference was that the about 0.5 percent iron has been added to the 70-30 cupro-nickel. This small amount of iron forms a more protective film. A similar amount of iron also has prevented impingement attack in the tests illustrated by Figure 14.

Rotating discs also can be used to show erosion corrosion of high copper alloys. Figure 18 shows a 5-inch diameter disc of Admiralty brass which was rotated horizontally in sea water with a tip speed of 25 ft/sec.[15] The appearance is quite different from that of the cast iron disc in Figure 7. In the case of Admiralty brass, attack is confined to the specimen's outer portion, possibly due to a metal ion cell or the removal of protective films. Both would work together to make the outer high speed portions of the disc anodic to the central portion.

Most of the attack on Admiralty brass occurred outside a radius of 1.75 inches

Figure 16—Impingement attack on low-iron 70-30 cupro-nickel alloy after jet test in sea water containing entrained air bubbles. Peak velocity was 12 fps.[21]

Figure 17—Absence of attack on high-iron 70-30 cupro-nickel alloy under same test conditions as Figure 16.[21]

or at speeds greater than 19 ft/sec. Average corrosion rate of the attacked portion was 305 mdd. The attack was uneven and localized with typical erosion pits. Maximum thinning occurred about 1.75 inches from the center rather than at the periphery, probably due to the current flow between the anodic and cathodic portions being greatest here. The decrease in thickness at the outer edge was 17 mils.

Figure 19 summarizes the results on disc specimens of 70-30 cupro-nickel. The attack depth is plotted against velocity in ft/sec. Velocity, of course, increases with distance from the center of the disc. The plot shows the abrupt increase in attack beyond a critical velocity. The critical velocity is higher for the high iron alloy. The high-iron alloy is attacked to a lesser extent below and above the critical velocity. In contrast to the behavior of Admiralty brass, the attack on the corroded portion of the 70-30 cupro-nickels was uniform. Average corrosion rates of the attacked portions were 220 and 290 mdd.

The advantage of having some iron present extends to alloys higher in copper and lower in nickel. Figure 20 shows results on 90-10 cupro-nickels. Iron content of these alloys ranged from zero to two percent. The high iron alloys are quite superior. Data were obtained on bars attached to a disc which rotated in a vertical plane. Two discs were used of different diameters so that two speeds were provided. These particular specimens were quenched to keep all the iron in solid solution. With other heat treatments the beneficial effect of iron in percentages over about one percent was less. Data on 70-30 cupro-nickels are included in Figure 20, and again the high iron alloy is superior. High iron 90-10 alloys compare favorably with the high iron 70-30 alloys.

An observation in these tests was that depth of attack varied with amount of air entrained in the solution. When the rotating wheel was near the surface of the tank, large volumes of air were whipped into the water. When the wheel was immersed deeply, less air was entrained. Excess air decreased the erosion corrosion. Some air is needed to depolarize the cathodes, but excess air evidently tended to keep the protective films in repair.

Erosion Corrosion of Other Alloys

Erosion corrosion occurs on other materials besides alloys of iron and copper, but the principles involved are the same. All the exposure conditions which determine corrosion rates under normal conditions will have some effect on erosion corrosion because they influence the nature and rate of protective film formation. Alloying which increases the corrosion resistance and develops more protective films will help reduce erosion corrosion. Erosion corrosion is likely to occur in pumps, valves, propeller blades, orifices, agitators, elbows, tees, heat exchanger tubes and other equipment in which high velocity water is encountered. Special testing devices may be needed to simulate special conditions.[24]

Figure 21 shows another example of erosion corrosion: a valve seat with deep grooves eroded in it. This attack was caused by wet steam. Water droplets impinging at high velocity removed protective films. The attack was quite local. Such attack is sometimes called wire drawing. The remedy is to find alloys with better and more corrosion resistant films.

Cavitation Erosion

Related to the effects of velocity is damage caused by cavitation erosion,[24]

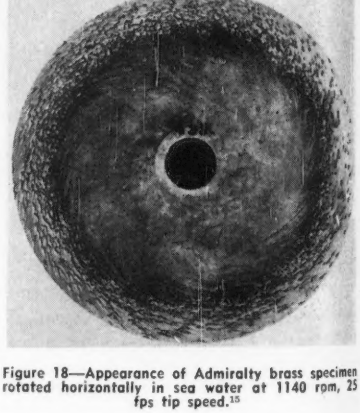

Figure 18—Appearance of Admiralty brass specimen rotated horizontally in sea water at 1140 rpm, 25 fps tip speed.[15]

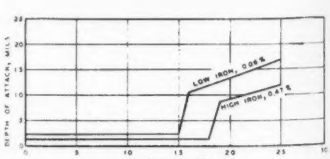

Figure 19—Effect of velocity on depth of attack of 70-30 cupro-nickel in sea water. Sixty-day tests at 26 C.[21]

which requires that the liquid be subjected to rapidly alternating ranges of pressure. At high relative motion between the metal and solution, pressure may be reduced locally so that boiling occurs. Small cavities of vapor form in the liquid. Return of pressure to normal causes an implosion as the cavities collapse. If a cavity is in contact with the metal, there is a high speed of liquid impact. The surface work hardens, roughens and

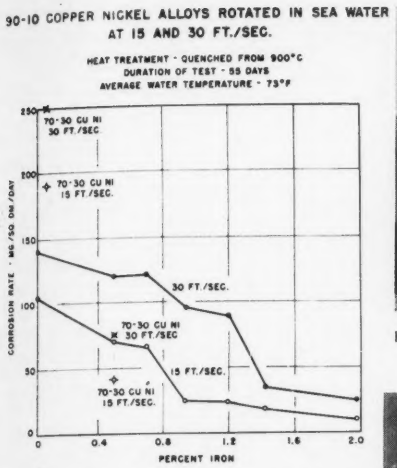

Figure 20—Effect of iron content on corrosion of 90-10 cupro-nickels rotated in sea water at 14 and 30 fps.[15]

cracks by fatigue. The result is a rapid, deep, spongy pitting.

This mechanism emphasizes the mechanical nature of the damage. Nevertheless, corrosion accelerates the destruction, possibly due to erosive effects removing protective films. Pitting and roughening of the surface by corrosion increase the damage. Conditions might be such that a corrosion resistant material would remain unaffected, whereas a material which pitted would cause turbulence that would lead to cavitation erosion.

Figure 22, an example of cavitation erosion, shows a cross section of a Diesel engine cylinder. Attack occurred on the cylinder's water side. The spongy nature of the attack is quite evident. Rate of attack was 3 inches per year, a rate too high to be accounted for by oxygen consumption, hydrogen evolution or stray currents.

Figure 23 shows another example of cavitation erosion which occurred on the low pressure side of a propeller blade. Hardness readings show the work hardening that occurred in the attacked area. Micro-examination of cross sections showed that the grains had been distorted and that fatigue cracks were present.

Cavitation erosion has been experienced in high speed pumps, hydraulic turbines, valves, near orifices and near obstructions. This erosion can occur wherever high velocity flow is encountered under low or medium pressure.

Jet, Venturi, and vibratory tests have been used to study cavitation erosion.[24] Results obtained by using the vibratory test method are summarized in Table 3.

Data show that both corrosion resistance and hardness are important in minimizing damage. Aluminum bronze and high tensile brass have similar mechanical properties, but aluminum bronze is superior to brass in corrosion resistance and suffers less cavitation erosion. High tensile brass is superior to the 85-5-5-5 bronze in mechanical properties and suffers less cavitation erosion than this bronze even though its corrosion resistance is inferior.

Figure 21—Wire drawing of valve seat by wet steam.[14]

Figure 22—Cross section of a Diesel engine cylinder showing cavitation erosion on the water side.[29]

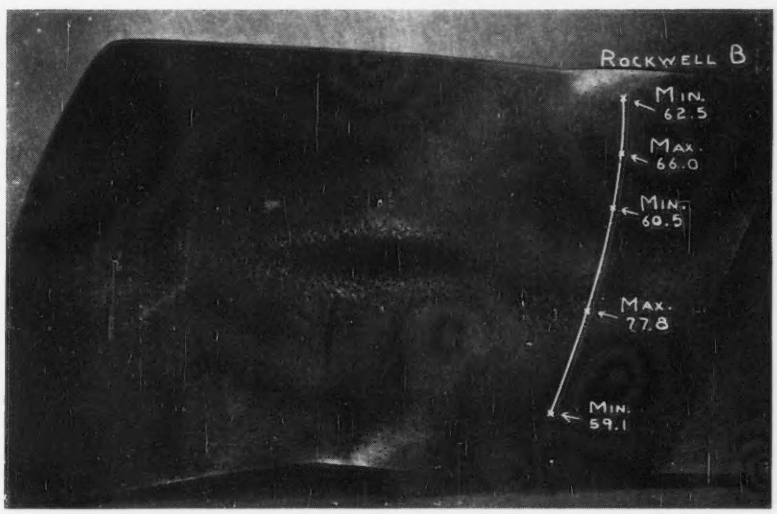

Figure 23—Cavitation erosion on low pressure side of a propeller blade.[24]

TABLE 3—Effect on Corrosion Resistance and Hardness on Cavitation Erosion of Cast Materials, Vibratory Test Method[30]

Alloy	Hardness BHN	Tensile Strength psi	WT. LOSS MG LAST 60 MIN.	
			Salt Water	Distilled Water
Aluminum Bronze	154	86,000	4.6	....
High Tensile Brass	156	85,000	13.9	....
Bronze, 85-5-5-5	62	30,000	25.0	24.3
Cu-Ni-Zn Alloy	155	73,000	9.8	....
Cu-Ni-Zn Alloy	187	103,000	7.0	....
Cu-Ni-Zn-Alloy	255	114,000	3.5	....
Cast Iron	214	30,000	46.9	30.2
Ni-Resist	179	33,000	15.5	5.8
18-8 Stainless Steel	168	.....	2.8	2.6

With alloys of the same type such as the copper-nickel-zinc alloys (in Table 3) with similar corrosion resistance, resistance to cavitation erosion increases regularly with hardness.

The importance of the corrosion is shown by comparing results in fresh water and in salt water. There is little difference between the behavior of stainless steel and bronze in the two media, but cast iron and Ni-Resist corrode more rapidly in sea water than in fresh water, and hence show greater cavitation erosion in sea water.

The influence of corrosion shows up in tests lasting only 60 minutes. The vibratory test produces severe damage in this short time and therefore is emphasizing the mechanical aspects of cavitation erosion. With the much longer times encountered in service, the influence of corrosion would be expected to be greater than indicated by Table 3.

Electrochemical Theory

A good way to understand the effects of velocity is to consider the electrochemical theory of corrosion. For several reasons, areas of different potential become established on the metal surface, and current flows between these areas. As the current flows, the cathodes and anodes polarize to about the same potential, limiting the total current and total corrosion.

At the anodes the polarization might be due to an accumulation of metal ions. At the cathodes the polarization might

be due to depletion in the supply of oxygen or hydrogen ions or to an accumulation of hydroxyl ions, depending upon the particular cathode reaction. Velocity removes the corrosion products and increases the supply of reactants, thus decreasing the polarization and increasing current flow and total corrosion.

Polarization also might be due to the formation of films at either anodic or cathodic areas. An increase in velocity may favor the formation of such films by bringing the reactants into closer contact with the metal. This will increase the polarization and decrease the current flow and total corrosion. High velocity and turbulence can erode away protective films and thus form anodic areas.

Determination of potentials and polarization curves is fundamental. These measurements explain all the effects of velocity and should help as each new problem is encountered.

Acknowledgment

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Any discussion of this article not published above will appear in the December, 1960 issue.

Techniques and Precautions

Corrosion Testing of Two Zirconium Alloys*

By STANLEY KASS

Abstract

Some of the external factors influencing the high temperature water and steam corrosion properties of two zirconium alloys have been investigated. These studies coupled with eight years of extensive corrosion testing of samples and reactor components have shown that surface preparation and the manner in which the corrosion test is performed greatly influences the resultant corrosion behavior. Cleaning, pickling, and rinsing of the metal surfaces, autoclave preparation, operation and cleanliness are described. Proper fixturing for the pickling and pressurized water testing are detailed.

6.3.20

Introduction

ZIRCONIUM alloys, because of their outstanding ability to withstand the corrosive attack of hot water and steam perform dual functions in pressurized water-cooled thermal reactors:

a. As cladding to prevent the access of the water coolant to the fissionable fuel and to prevent the access of the fission products from entering the plant

b. As a structural member to support the reactor core and to prevent distortion as a result of fission processes.

These excellent corrosion properties, characterized by the formation of a hard, tightly-adherent film of monoclinic ZrO_2 are inherent in the alloys. However, the behavior of samples and components in the hot aqueous media is greatly influenced not only by heat treatment, metallic or gaseous contamination, variations in alloy and impurity contents, etc., but also by the condition of the surfaces prior to corrosion exposure and the manner in which the corrosion test is conducted.

The inherent corrosion properties of the alloys, designated here Alloys A and B* as well as the "internal factors" (heat treatment, contamination, etc.) influencing the aqueous corrosion have been extensively reported.[1-7] For the purpose of review, the corrosion behavior of Alloy A is summarized in Figure 1 where the total cumulative weight gain per unit of original area is plotted versus the total exposure time on logarithmic scales. The data points from which the curves were drawn are averages of greater than 100 samples from various lots and the deviation between individual samples is no larger than 15 percent of the mean value. The curve for the corrosion in 750 F degassed steam at 1500 psi shows a change in slope at a weight gain of 40 mg/dm² in the vicinity of 40 days. It is noted in Figure 1 that in 680 F degassed water, an inflection in the log weight gain-log time curve normally occurs at a weight gain of approximately 35 mg/dm² after an exposure of about 115 days. No inflection or slope changes are observed in the 600 or 550 F tests, evidently because very long exposures would be required to reach the weight gains at which the change in slope occurs at the lower temperatures. The corrosion rates for Zircaloy-2 are constant after "transition." The rates are 1.1 mg/dm²-day at 750 F, 0.37 mg/dm²-day at 680 F and 0.06 mg/dm²-day (extrapolated value) at 600 F.[2]

A comparison of the corrosion properties of Alloy A and Alloy B is shown in Figure 2 which is presented in the same logarithmic manner as Figure 1. It should be noted in Figure 2, that within 40 days[1] exposure in 750 F steam, Alloy B is indistinguishable from Alloy A. However, the "post-transition" corrosion rate for Alloy B is lower by about 20 percent than that of Alloy A.

The influence of the "external factors" or testing methods on the aqueous corrosion, although of great interest to those concerned with the fabrication, purchasing and quality evaluation of zirconium alloy materials and components or zirconium alloy development, has received little coverage in the literature.

This report will discuss principally the "external factors" influencing the high temperature water and steam corrosion behavior of Alloys A and B. Although a majority of the items discussed in this summary are the results of many laboratory bench-type studies over the past six to eight years, similar beneficial or de-

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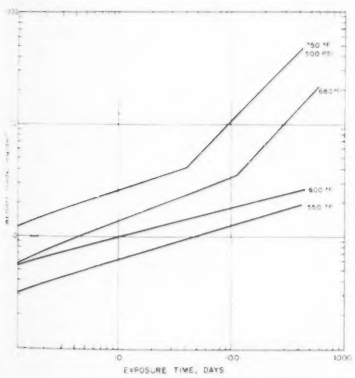

Figure 1—Weight gain-time curves for the corrosion of zirconium alloy A in high temperature water and steam. 750 F, 680 F and 550 F specimens annealed below 800 C; 600 F specimens as hot rolled.

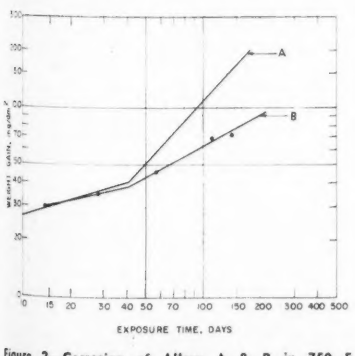

Figure 2—Corrosion of Alloys A & B in 750 F steam (1500 psi). Specimens furnace cooled from 775 C. Alloy B, average of 4 samples from each of 16 ingots.

* Nominal composition: Alloy A (Zircaloy 2)—1.5 tin; 0.12 iron; 0.10 chromium; 0.05 nickel; max 60 ppm nitrogen. Alloy B (Zircaloy 3)—0.25 tin; 0.25 iron; bal Zr.

About the Author

Kass

STANLEY KASS—He has been with Westinghouse Electric Corporation since 1946, having transferred to the Bettis Atomic Power Division in 1952 where he now serves as Lead Engineer, Corrosion Section, Nuclear Core Department. He received a BS in Chemistry from Ohio University in 1943. Mr. Kass' principal field of work is in the corrosion behavior of nuclear reactor materials including zirconium, uranium, hafnium and their alloys.

TABLE 1—Typical Flow Diagram for Corrosion Testing

1. Fabricate and identify specimens or components.
2. Detergent or solvent clean.
3. Suspend on pickling fixture.
4. Rinse in hot tap water.
5. Etch in HF-HNO_3-H_2O.
6. Immediately transfer to flowing tap water and rinse.
7. Rinse in a second tap water tank.
8. Rinse in high purity water.
9. Dry.
10. Inspect (repickle, if necessary).
11. Measure surface area and weight if quantitative data are sought.
12. Fix specimens or components on fixture.
13. Load fixture into autoclave.
14. Add required amount of high purity water to autoclave.
15. Seal autoclave and activate auxiliary equipment.
16. Vent off dissolved gases.
17. Measure time when test conditions are achieved.
18. At conclusion of exposure time, deactivate auxiliary equipment.
19. When cool, open autoclave and remove fixture.
20. Rinse in high purity water.
21. Dry.
22. Weigh coupons and calculate weight gain per unit area if quantitative data are required.
23. Inspect.

leterious effects are noted whether coupons 1 in. x 1 in. x 0.1 in. or components 6 in. x 6 in. x 120 in. are employed.

The Corrosion Testing Procedure

Summary

In general, the corrosion testing of zirconium alloy coupons and components includes surface cleaning, pickling, rinsing and autoclaving. A typical flow diagram of the procedure is shown in Table 1. Many of the operations appear lengthy, involve considerable capital investment and require great dexterity and specific precautions. Experimental laboratory results leading to the establishment of the process as well as the specific precautions and techniques which must be employed in order to obtain meaningful corrosion behavior will be discussed in detail.

Sample Preparation

The amount of surface metal to be removed from mill product surfaces is generally dependent upon the source and history of the material. Sufficient material must be removed by machining from all stock to eliminate all surface effects. (This recommendation is not valid when investigations which require evaluation of the original surfaces are made.)

Specimen or component surfaces must be free of all oxide, cracks, voids, fissures, pits, streaks, blisters and other surface defects. Items exhibiting these defects should be repaired or rejected.

Identification can be made by any means which does not cause oxide formation or contaminate the surface with foreign metal. Methods which have been successfully applied are vibra-tooling, steel stencil stamping and engraving. (Identification marks should be large enough to read but sufficiently small so as not to cover the entire specimen surface. Numbers 1/16 or 1/8 inch high have been used successfully.)

The corrosion properties of the alloys are not influenced or dependent upon specimen or component size or shape; hence, no formal rules or requirements have been established. The following recommendations are made, however, to insure that meaningful and quantitative corrosion data are obtained when required:

a. The specimens should be rectangular in shape and square on all corners to facilitate surface area measurements.

b. Specimen surfaces should be machined to a 63 rms finish or better with a sharp tool, avoiding smearing or dragging of the metal, to insure the measured surface area is a close approximation of the actual area.

c. The specimens should have a hole at one end to facilitate handling in cleaning, pickling and autoclaving. Diameter of the hole should be adjusted so that the surface area of the cylinder forming the hole approximates the surface area of metal removed to make the hole.

Surface Cleaning

All dirt, grease, oil, fingerprints, etc., must be completely removed from the specimens or components by detergent washing or solvent cleaning before chemical etching. Surface contamination by any of these will result in preferential attack by the etchant.

Cleaned items must be handled with clean, lint-free gloves to prevent surface contamination. Surfaces of all items which come in contact with cleaned specimens or components must be cleaned in a manner equivalent to that used on specimens.

Cleaned parts, when not being processed, should be enclosed in a clean container or wrapped in either heavy paper or plastic to prevent surface contamination.

Etching

The corrosion rates of zirconium and its alloys are affected by the condition of the surfaces induced by machining, abrading or chemical contamination. A truly representative surface on good quality material permits one to obtain a protective oxide, while a poor surface on the same material results in loose white oxide. Because of the sensitivity of the zirconium alloys in this respect, great care must be exercised in surface preparation if spurious results are to be avoided. Mechanical operations, such as machining and abrading, on the surface of zirconium alloys give rise to a layer of disturbed metal at the surface and for an appreciable depth under it. This disturbed layer exhibits a more rapid corrosion rate than does the bulk metal, so it must be removed before corrosion exposure. This effect is illustrated in Figure 3 which correlates the extent of the disturbed metal under a machined surface as revealed by X-ray line broadening measurements with the appearance of zirconium specimens after corrosion. The incidence of white corrosion, indicating accelerated attack, is seen to correlate with the extent of disturbed metal.

The recommended process for the etching of Alloys A and B is discussed fully below.

Previously cleaned specimens should be suspended individually on clean, previously pickled Type 18-8 stainless steel hooks. When mounting holes are not desirable or feasible, special fixtures must be designed to minimize the contact of Zircaloy with stainless steel. Figure 4 demonstrates the preferential attack which may occur during etching when Zircaloy is in intimate contact with the stainless steel support.

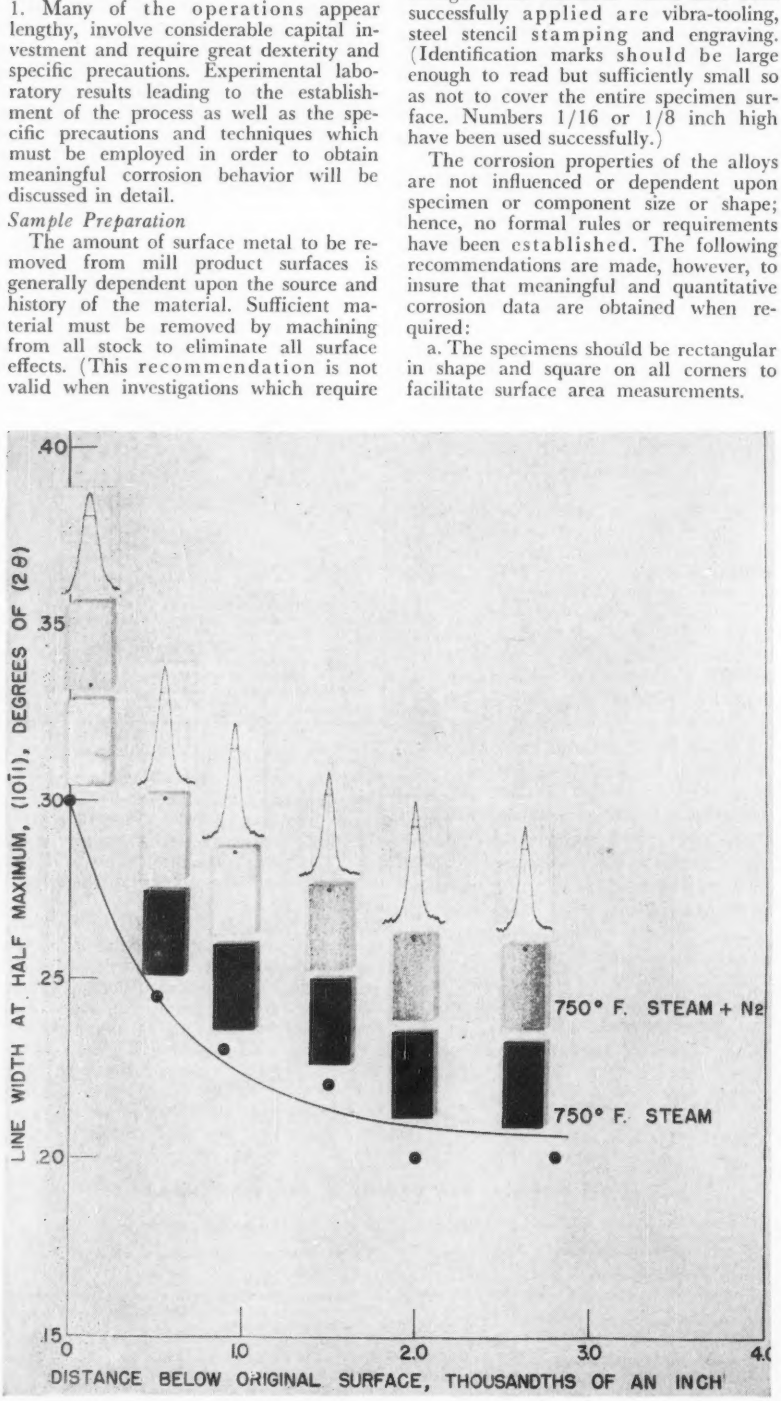

Figure 3—Effect of surface condition on the corrosion of zirconium.

Before etching, the items should be rinsed in 120 F tap water for one to two minutes to insure a more rapid wetting by the acid etchant.

Etching baths containing 20 to 50 v/o nitric acid (1.42 sp. gr.) and 2 to 5 v/o hydrofluoric acid (52 percent) have been used for the efficient removal of surface metal. Dissolution rates of the zirconium alloys vary with acid concentrations and bath temperature. A comprehensive review of the dissolution rates may be found in Reference 8. As a general rule, however, temperature of the pickling bath should be maintained between 80 and 120 F, preferably at the lower limit. Items pickled at temperatures in excess of the upper limit result in pitted and blistered surfaces, those pickled at temperatures below the lower limit require excessively long immersion times and exhibit non-specular or dull surfaces. Furthermore, difficulties which are discussed in the next section may be encountered when items are pickled at temperatures greater than 120 F. A large ratio of etchant volume to component surface or an outershell of cooling water facilitates the maintenance of the temperature limits, dissipating or removing the heat generated by the exothermic zirconium-acid reaction.

The addition of many surface active compounds stable in the hot acid solution tend to lower the surface metal dissolution rate dependence upon temperature, but do not facilitate the removal of residual etchant from the surfaces.[9]

For the 39 percent nitric-3.5 percent hydrofluoric acid solution, approximately ½ lb. of metal per gallon of etchant may be dissolved before the bath need be changed. If it is assumed that two mils of metal are removed per surface, a total of 500 sq. in. of metal may be dissolved per gallon of solution before the etchant need be discarded.

The components and the etchant must be agitated throughout the pickling operation to prevent preferential attack (ridging, erosion, or pitting). Optimum surfaces for corrosion test can be obtained by using either of the following operational techniques:

a. The fixture and items are continuously and rapidly raised and lowered through the etchant. The items break the liquid surface upon raising and lowering. This technique yields bright specular surfaces and results in bright temper films after corrosion tests.

b. The parts are totally immersed in the etchant which is agitated by a fine stream of filtered air bubbles impinging upon the components. This technique also results in bright specular surfaces.

When large components are to be etched, a combination of the above two techniques has yielded bright specular surfaces.

Rinsing

Contamination of the surfaces by the $HF-HNO_3-H_2O$ etchant occurs when the etchant is not thoroughly rinsed or when the etchant is permitted to dry on the surfaces. In the latter case, the residue is insoluble in water. Inadequate rinsing, or etchant dried on the surface, leads to the formation of loose white corrosion product when exposed to high temperature water.

Figure 5 illustrates the corrosion behavior of Alloy A after proper and improper etching techniques. All specimens were corrosion tested in 680 F water after two mils per surface were removed by $HF-HNO_3-H_2O$ etching. The first set was tested after the specimens were thoroughly rinsed in water. Note the black oxide surfaces with no evidences of white corrosion product. The second and third sets of specimens were tested after:

a. The etchant was permitted to dry on the specimens which were subsequently scrubbed with water and a soft brush.

b. The etchant was permitted to dry and no cleaning attempts were made.

Further dramatic evidence that corrosion resistance can be markedly deteriorated by improper etching techniques is shown in Figure 6, a photograph of zirconium test specimens which were clamped together and exposed for seven days at 650 F in water or steam containing 200 ppm of sodium fluoride. Extremely severe corrosion is noted under these conditions at mating surfaces.

A more frequent difficulty in the corrosion testing of zirconium alloys is not the case of heavy white spalling oxide, but the more subtle effects involving only weight gain values or slightly milky surfaces (Figure 7). This condition is brought about during the transfer of the items from the etchant to the rinse bath.

a. Although the pickling operation may have been performed properly and the etchant temperature was below the maximum limit, if the transfer is not made rapidly, the pickling acid clinging to the surfaces becomes spent. The residual reaction product is then deposited upon the surfaces.

b. If the pickling operation was performed properly but in excess of the

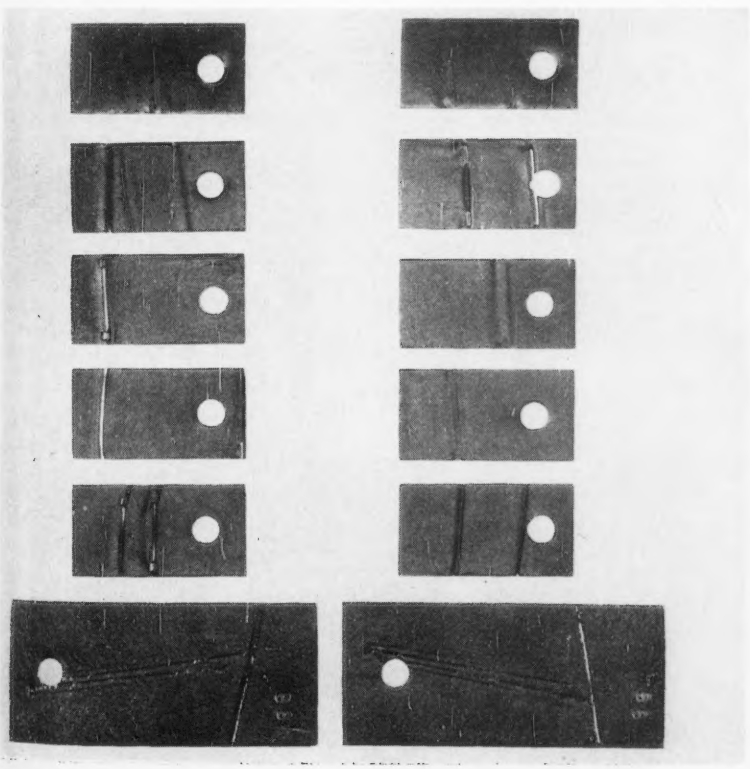

Figure 4—The effect of intimate contact between Alloy A and stainless steel upon etching.

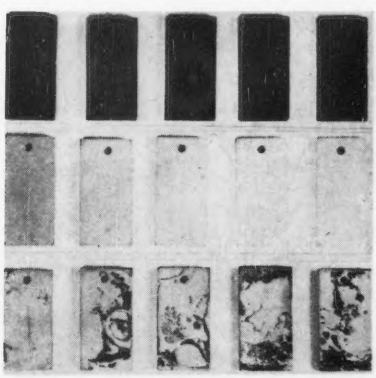

Figure 5—Specimens not rinsed after etching but scrubbed before corrosion testing. Effect of rinsing and washing on the corrosion of Alloy A specimens tested in 680 F water for 58 days. Top—Rinsed immediately after etching. Middle—Not rinsed after etching. Bottom—Not rinsed after etching but scrubbed before corrosion testing.

specified temperature range, the residual pickling acid clinging to the surfaces becomes spent almost immediately due to the residual heat of the component and deposits upon the surfaces.

The following rinsing procedure has been successfully utilized at the Bettis Plant for the past several years:

a. The components and specimens are immediately and rapidly transferred from the etching solution to the first rinse tank containing flowing tap water and allowed to rinse a minimum of five minutes or until all traces of acid entrapment and contamination are removed (pH paper checks). The bath size and water flow rate are adjusted to attain a rate of flow equivalent to one bath change per minute. (If the bath size and/or flow rate requirements cannot be met, additional rinse time or rinsing tanks are used to eliminate the carryover of acid contamination.)

b. The specimens or components are then transferred to a high purity water* rinse tank to remove the final traces of contamination from the fluoride etchant or the tap water. This final rinsing is attained by either of the following methods:

1) Dynamic

The parts are immersed in a tank containing flowing hot (180 F minimum) water. The inlet water meets high purity requirements. The total immersion time is the time necessary for the effluent water to meet the following requirements plus 5 min: pH, 7.0 ± 1.0; resistivity, 100,000 ohm-cm (minimum).

2) Static

The items are immersed in a tank containing static boiling high purity water for a minimum of 5 min. The bath is changed whenever the water exceeds the minimum specification already set forth and additional rinses are performed until the bath meets specification.

Drying

The parts, after removal from the rinse tanks, should be dried by any suitable means which provides for free access of air to the surfaces. Air drying, ethyl, alcohol rinsing and wiping with clean lint-free cloths all have proved satisfactory.

Post-etching Inspection

Properly etched surfaces are bright, shiny and exhibit no surface irregularities. A well-etched surface has a metallographic finish; i.e., the microstructure can be seen fairly well under a microscope. It should be noted that post-pickling observations will not always point out faulty pickling and rinsing operations. Improper techniques will be dramatically pointed out at the conclusion of the corrosion test (erroneous weight gain values, white areas, or generally cloudy surfaces). The items should be inspected for stains, areas of preferential attack and luster. Items which exhibit areas of preferential attack must be refinished and repickled. Items exhibiting stains or dull surfaces must be repickled.

Weighing and Measuring

After drying, the specimens being used for quantitative corrosion measurements should be stored for a minimum of two hours in a clean, dry area at the same humidity as the balance to permit the specimens to equilibrate with the balance.

The specimens should be weighed to a tolerance of ± 0.2 mg using a balance capable of being calibrated to a tolerance of ± 0.10 mg. The balance must be calibrated at least once each day and zeroed for each five weighings.

The specimens should be measured to a precision of ± 0.008 cm.

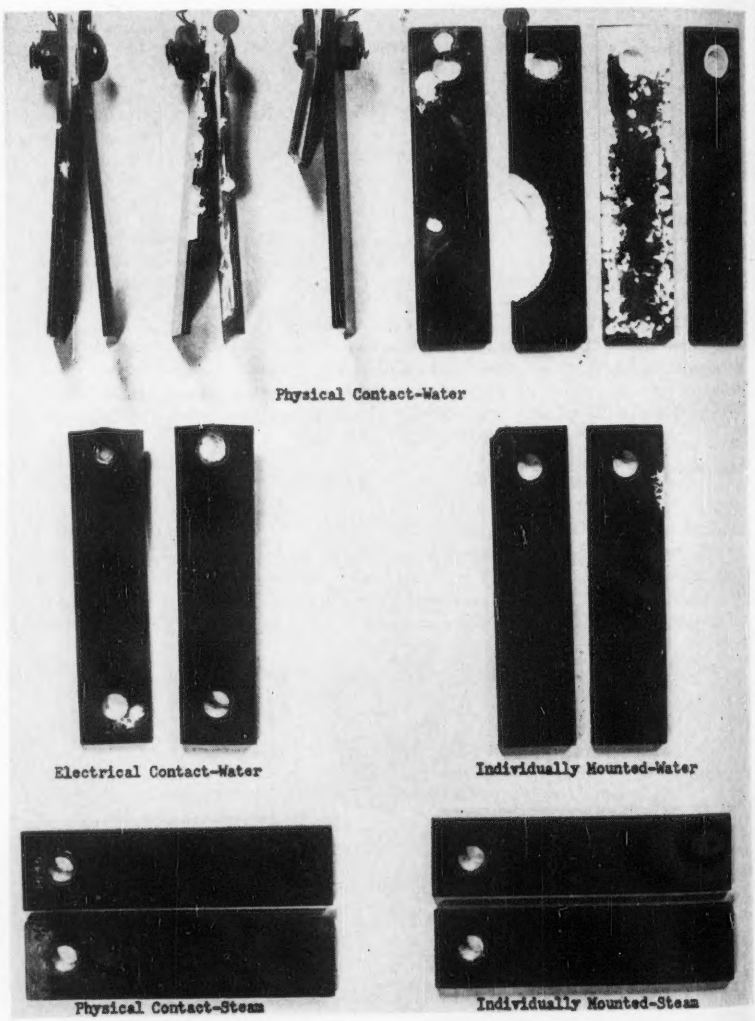

Figure 6—Corrosion of zirconium in 650 F water and steam containing 200 ppm sodium fluoride—four days exposure.

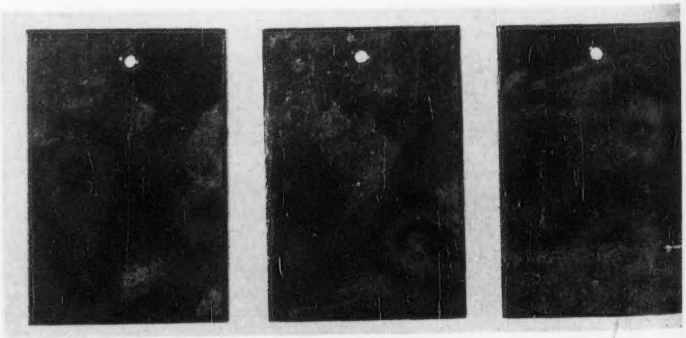

Figure 7—Typical surfaces after corrosion testing showing insufficient rinsing after etching.

* High purity water as defined here meets the following requirements: pH, 7.0 ± 1.0; Resistivity, 500,000 ohm-cm (minimum).

Autoclaving Equipment

It is recommended that the autoclaves used be ASME Code stamped for not less than 3000 psi maximum safe working pressure and 850 F safe operating temperature in accordance with Section 8 of the ASME Boiler and Pressure Vessel Code. They should be constructed of an ASME approved corrosion resistant steel.

Fixtures

The specimens should be suspended on stainless steel "trees" (Figure 10), during corrosion exposure, fabricated by spot welding AISI Type 316 or 347 stainless steel wire to stainless steel rods every two inches. Stainless steel washers are used as spacers to prevent positive contact between the specimens. Specially designed fixtures should be used to support components during corrosion test thus minimizing specimen-to-fixture contact.

All new and reworked fixtures and jigs should be cleaned, passivated in nitric acid, rinsed in high purity water and autoclaved for one day in 750 F steam or equivalent before use. The fixtures and jigs must be checked for corrosion; fixtures or jigs exhibiting loose corrosion product may not be used for corrosion testing.

Temperature control should be maintained at the autoclave heating units while the temperature in the autoclave, measured in the thermocouple-well, should be recorded on a multi-point temperature recorder.

Pre-test Requirements

Before specimens may be tested in a new or reworked autoclave, the equipment must be thoroughly cleaned in order to minimize water contamination. The operations should include a detergent scrub, two to three tap water rinses, ethyl alcohol wiping and rinsing with at least two complete changes of hot (160 minimum) high purity water.

Each new or reworked autoclave must be qualified or rechecked by testing a minimum of three coupons (fabricated from a lot of previously tested material which has satisfactorily passed the requirements for weight change) at 680 F for 14 days in high purity water. The coupons may not exhibit stains, pits or areas of general corrosion attack and must exhibit surfaces comparable to those coupons which were tested in a qualified autoclave.

The temperature gradient at each operating temperature must be measured in the vertical direction within the testing volume for each autoclave. This check must be made before a new or reworked autoclave is placed in use and at least once every four months after it has been

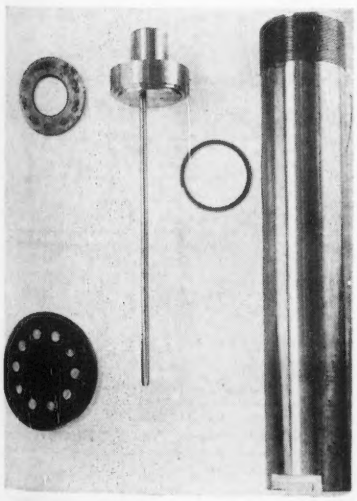

Figure 8—Expanded view of typical autoclave.

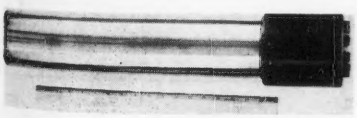

Figure 9—Assembled autoclave.

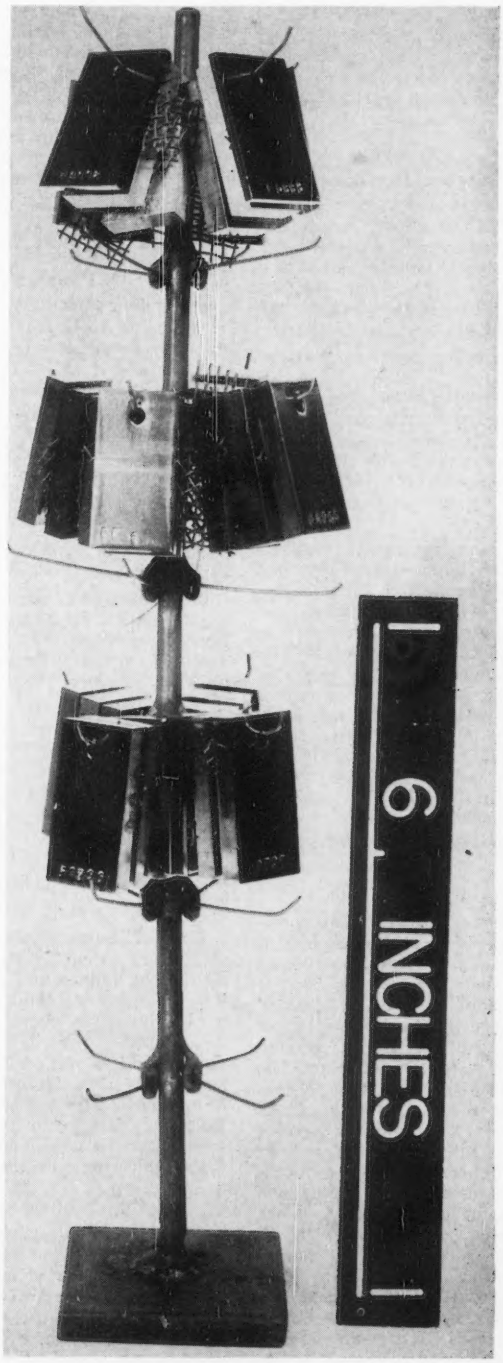

Figure 10—Typical tree and specimens before insertion into the autoclave.

placed in use. The useful coupon testing volume should then be restricted to that volume which corresponds to nominal temperature ± 5 F. Nominal temperature ± 10 F may be permitted when large components are tested.

Autoclave Loading

The fixture supporting the components to be autoclaved, or the "tree" containing specimens and stainless steel washers alternately suspended on the "arms," should be placed within the useful testing volume of the autoclave as determined by the temperature gradient study above. Immediately before placement into the autoclave, all fixtures, jigs, coupons, components should be rinsed for a minimum of 30 minutes in 180 F high purity water.

Water Purity and Degassing

High purity water is the recommended medium for all simulated service or quality determination corrosion tests. This water, which initially exhibits a specific resistivity greater than 500,000 ohm-cm and is readily obtained by mixed-bed demineralizing, is desired to:

a. Simulate reactor plant operating conditions.

b. Eliminate spurious results attributable to dissolved or transported impurities present in tap water.

c. Serve as a reproducible medium.

d. Prevent excessive corrosion of the stainless steel autoclaves, parts and fixtures.

Although oxygen and nitrogen concentrations of a few hundred milliliters per kilogram of water do not cause excessive corrosion of zirconium alloys, the high purity water should contain no more than 0.03 cc O_2/kg of water. The low oxygen content prevents excessive corrosion of the stainless steel parts and subsequent deposition on the test components, rendering evaluation of the corrosion test difficult due to masking effects. Many procedures have been developed which can furnish water meeting the above requirements. Three of these procedures are presented and discussed here.

Details of Water Tests

1. The fixture containing the specimens or component is loaded into a clean, dry autoclave. High purity water having a resistivity greater than 500,000 ohm-cm and a pH of 6 to 8 is added to the autoclave. The amount of water added is calculated so that 80 percent of the net volume (autoclave volume less component and fixture volume) will contain liquid at the temperature of the test (see Table 2). (The 20 percent vapor space is allowed for safety.) In addition, a 10 percent excess of water is added. The autoclave is sealed and the heating units are energized. When the internal temperature reaches 250 C, the vent valve is opened to the atmosphere or venting manifold for three minutes. The vent valve is closed and 10 minutes later again opened for three minutes and closed for duration of test.

2. The fixtures and components are loaded into the autoclave as above and the required volume of high purity water is added. The autoclave, except for the venting valve, is sealed and the heating units are energized. After the internal water temperature has reached 180 to 210 F, the temperature will rise very slowly due to the boiling action. The vent valve is closed approximately eight minutes after this semi-steady state is reached.

3. Degassed high purity water is prepared and stored in a separate closed system utilizing a steam-scrubbing degassing column or equivalent. The required amount of water (without the 10 percent excess) is added to the autoclave using a positive placement pump, solenoid time-operated valve, a vacuum pump and an inert gas pressurizer. The autoclave is evacuated to approximately 10 microns Hg pressure, backfilled with helium or argon and re-evacuated. The autoclave is pressurized to 5 psig with helium or argon and the required amount of high purity water is added.

750 F Steam Tests

a. The fixture containing the items to be tested is loaded into a clean, dry autoclave as above. Sufficient high purity water is added to fill the autoclave ¾ full. This large quantity of water is added to decrease the initial free air space. The autoclave, except for the venting valve, is sealed and the heating units are activated. When the temperature reaches 600 F, the vent valve is closed. As the temperature continues to rise, the pressure is maintained at 1500 psi by momentarily opening the vent valve.

b. A procedure similar to that of water tests, (3), is used (addition of degassed water to the autoclave through a closed system) except that the pressures in excess of 1550 psig are vented to the atmosphere.

Test Tolerances

Effects of temperature and pressure on the corrosion behavior of zirconium and its alloys studied at Bettis and at Nuclear Metals, Inc. have shown that corrosion increases with increasing temperature and time but varies sporadically with pressure.[10,11,12] The test tolerances are given in Table 3.

It should be noted when the corrosion test serves as an inspection tool only, all time at temperature and pressure below the tolerance limits may be disregarded but time above the limits may be accepted as time in test. These deviations should not be permitted when corrosion rate data (coupon tests) are to be obtained. The test time is measured from the time when the nominal test temperature is reached until the time when the heaters are de-energized.

Post-Test Operations

After the test period, the autoclave heaters should be de-activated. The autoclaves must be cooled to room temperature within 18 hours of de-energizing the heaters.

After the autoclave is unsealed, the fixtures are removed from the vessel. The items are removed from the fixtures, rinsed in high purity water, dried by any suitable means which provides for free access of air to the surfaces and inspected.

After drying, specimens should be stored for at least two hours in a clean, dry area at the same humidity as the balance. The specimens should be weighed as before and the weight change per unit area (mg/dm²) calculated.

After data reporting, the test may be discontinued or continued for extended corrosion exposure. Parts included in programs for extended exposure should be returned to the autoclaves without disturbing the surfaces or removing the oxide film.

Conclusion

The excellent corrosion properties of the zirconium alloys tested are greatly influenced by surface preparation and the corrosion test procedures. Meaningful corrosion data or inspection can be obtained only through careful preparation and strict control of the corrosion test.

Acknowledgments

The author acknowledges the work of the various corrosion testing laboratories at the Bettis Plant, Dr. K. M. Goldman, Messrs. F. J. Lennon, W. Heineman and H. E. Himes for initiating and following many of the corrosion investigations. Thanks are due Dr. D. E. Thomas for many helpful comments and discussions.

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Table 2—Computation of Volume of Water

The required quantity of water can be computed as follows:

Volume H_2O, ml = K (Vo—Vs)

V_o = Autoclave volume, ml

V_s = Total sample and fixture weight, g

7

Temp. °F	K
300	0.7348
500	0.6329
550	0.5954
600	0.5550
650	0.4980
680	0.4489

Table 3—Test Tolerances

COMPONENTS (When Corrosion Data Not Required)		
	Nominal	
Tests	Pressure psi ±	Temp. °F ±
Steam	100	10
Water	200	10
COUPONS (When Corrosion Data Required)		
Steam	100	5
Water	100	5

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DISCUSSION

Question by Norman D. Groves, Reading, Pa.:
Have you had an opportunity to demonstrate the relationship between laboratory results and actual field experience?

Reply by Stanley Kass:
Yes. The same precautions are required in laboratory work as in field work.

Question by Warren E. Berry, Battelle Memorial Institute, Columbus, Ohio:
It was said that during pickling zirconium samples must be raised from the solution or an air jet must impinge on the zirconium when it is completely immersed in the pickling solution. What effects are observed if these precautions are not observed?

Reply by Stanley Kass:
No deleterious effects upon the resultant corrosion are noted if the techniques described above are not followed. The procedures, however, produce bright, specular surfaces which facilitate the inspection of the items both before and after corrosion exposure. Surface defects, surface contamination by residual etchant, etc. are more readily discernible when the background is bright and lustrous. Secondly, the luster of the oxide film initially formed may be used as a qualitative measure of corrosion. If the underlying metal surface is non-specular, dull oxide films are produced and may lead to erroneous conclusions about the corrosion properties of the component.

Any discussion of this article not published above will appear in the December, 1960 issue.

ERRATA

High Temperature Metal Deterioration In Atmospheres Containing Carbon-Monoxide and Hydrogen by W. B. Hoyt and R. H. Caughey. Corrosion, Vol. 15, No. 12, 627t-630t (1959) Dec.

On Page 629t four captions line were incorrectly placed. Please rearrange the following complete caption lines:

Figure 8 line in spot now occupied by Figure 9 line

Figure 9 line in spot now occupied by Figure 11 line

Figure 10 line in spot now occupied by Figure 8 line

Figure 11 line in spot now occupied by Figure 10 line

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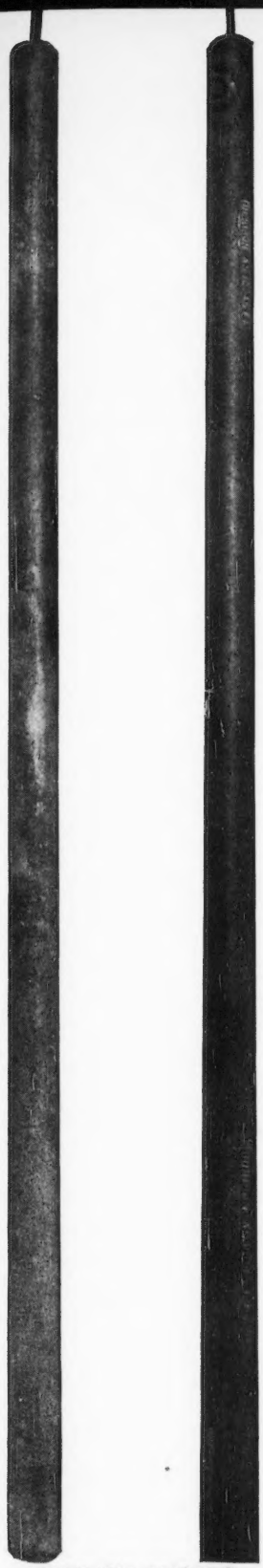

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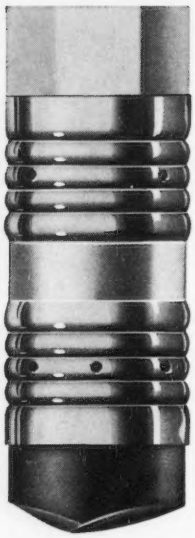

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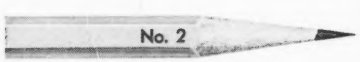

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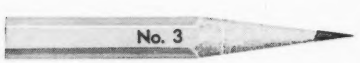

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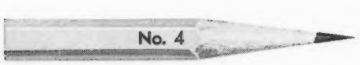

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